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# **A Novel Combination of Micromechanical Testing And Thermal Analysis**

*to Investigate the Temperature Dependence of Interfacial  
Adhesion in Fibre Reinforced Polymer Composites*

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University of Strathclyde  
Glasgow, Scotland*

**[www.strath.ac.uk/compositematerials/](http://www.strath.ac.uk/compositematerials/)**

# Thanks to



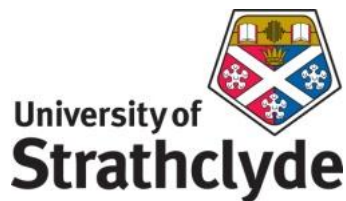
**Glasgow Research Partnership in Engineering  
- funding**



**Owens Corning – glass fibres**



**SABIC - polypropylene**

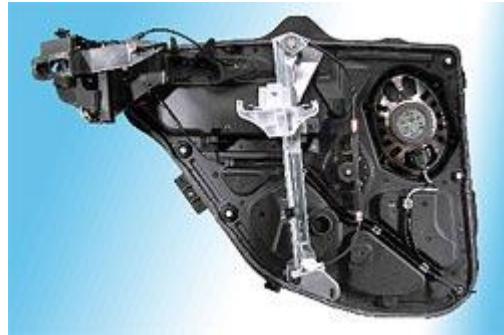


**Liu Yang - measurements**

# Outline: TMA – Microbond Testr

- **Introduction Thermoplastic Composites**
  - Residual Thermal Stresses
  - Glass Fiber-Thermoplastic IFSS
- **IFSS Experiments**
  - Conventional Microbond Test
  - Microbond Test in the TMA
- **Results**
- **Conclusions**

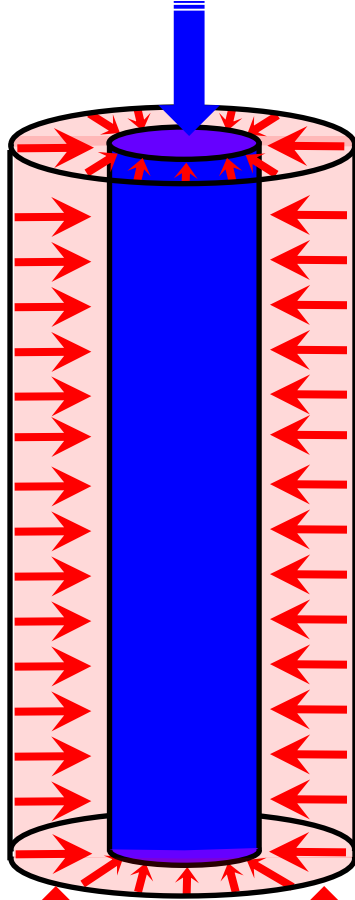
# Thermoplastic Composites Technology



- Strong continuing growth
- Attractive Performance:Price ratio
  - “Clean” processing - no chemistry
  - Intrinsically recyclable
- Still room for improvement
  - particular need to better understand (and increase) GF–PP interface stress transfer capability (IFSS)
- Processed mostly by moulding

# Residual Stress at the Interface

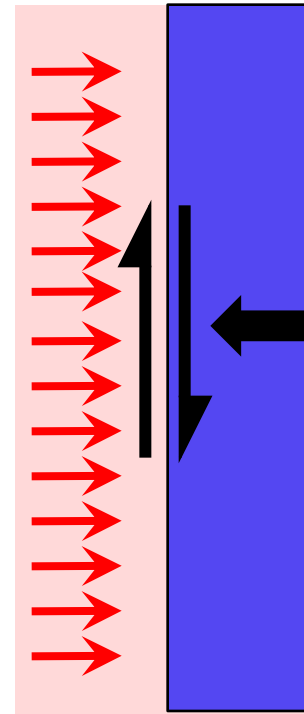
**Fiber – Low LCTE ( $\alpha$ )**



Large  $\Delta\alpha$   
and  $\Delta T$  from  
processing to  
room temp  
results in  
radial  
compressive  
stress at the  
interface

**Polymer – High LCTE ( $\alpha$ )**

$\mu_s$  – coefficient of static  
friction at interface



Interfacial  
stress transfer  
capability  
from residual  
radial stress

$$\tau = \mu_s \sigma_r$$

$\sigma_r$  – radial  
compressive stress

# Modelling Interfacial Residual Stress

$$\sigma_{rm} = E_m (\alpha_m - \alpha_{fT}) \Delta T$$

$$\sigma_{rm} = A_1 (1 - b^2 / r^2) \text{ where } b = F(V_f)$$

$$\text{for } A_1 \text{ solve } \begin{bmatrix} X_{11} & X_{12} \\ X_{21} & X_{22} \end{bmatrix} \begin{bmatrix} A_1 \\ A_3 \end{bmatrix} = \begin{bmatrix} (\alpha_m - \alpha_{fL}) \Delta T \\ (\alpha_m - \alpha_{fT}) \Delta T \end{bmatrix}$$

$$\text{where } X_{11} = 2 \left( \frac{\nu_m}{E_m} + \frac{\nu_A}{E_L} \frac{V_m}{V_f} \right) \quad X_{12} = - \left( \frac{V_m}{E_L V_f} + \frac{1}{E_m} \right)$$

$$X_{21} = - \left( \frac{(1 - \nu_f) V_m}{E_m V_f} + \frac{(1 - \nu_m)}{E_m} + \frac{(1 + \nu_m)}{E_m V_f} \right) \quad X_{22} = \frac{X_{11}}{2}$$

Nairn, J.A., Polymer Composites, 6, (1985) 123.

Wagner H.D. and Nairn J.A., Compos.Sci.Tech., 57, (1997) 1289.

# Residual Thermal Stress at Interface

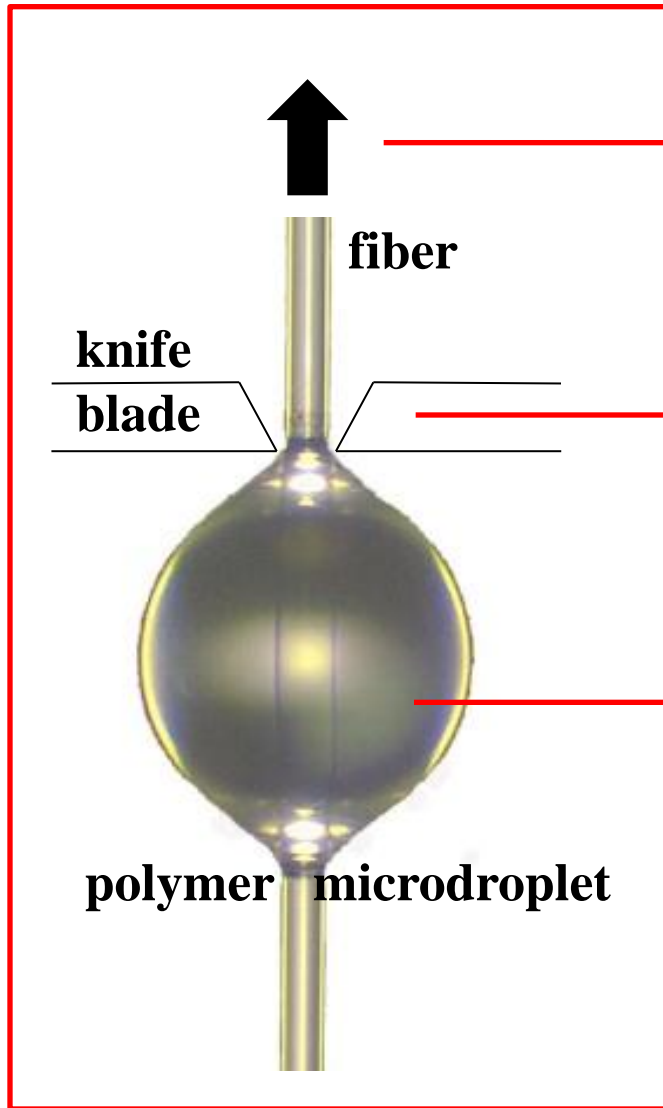
In both models  $\sigma_{rm} \propto \Delta T \Rightarrow \underline{\text{IFSS} \propto \Delta T}$

Hence the question –

is the apparent interfacial strength (IFSS) in thermoplastic composites temperature dependent ?



# Microbond Test for GF-PP IFSS



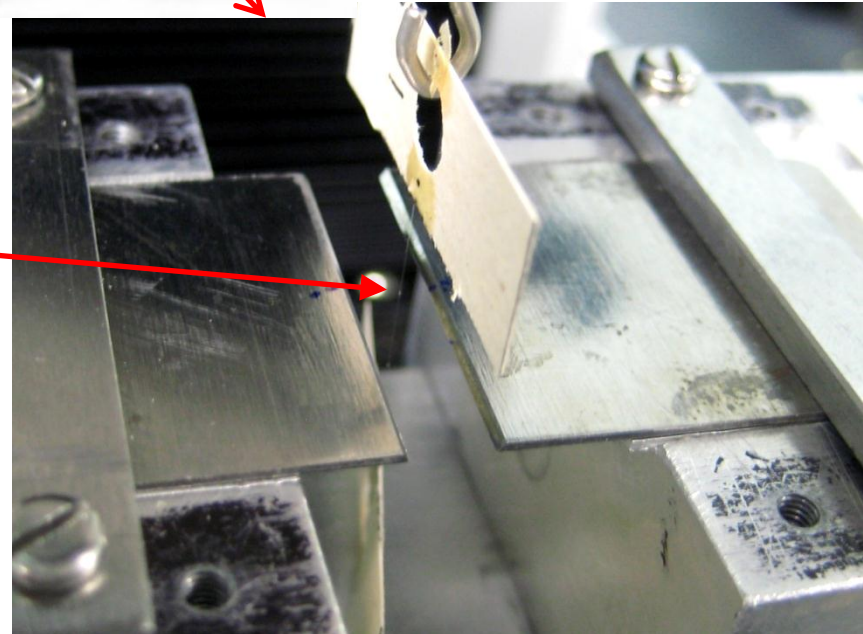
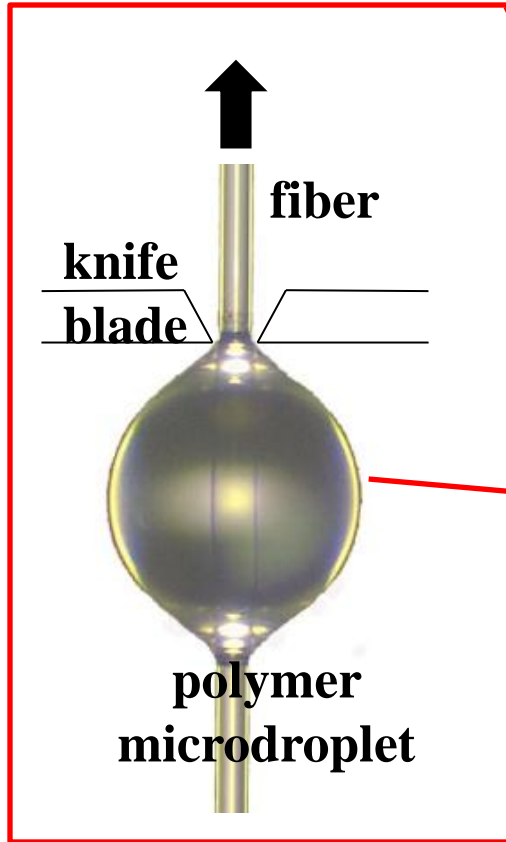
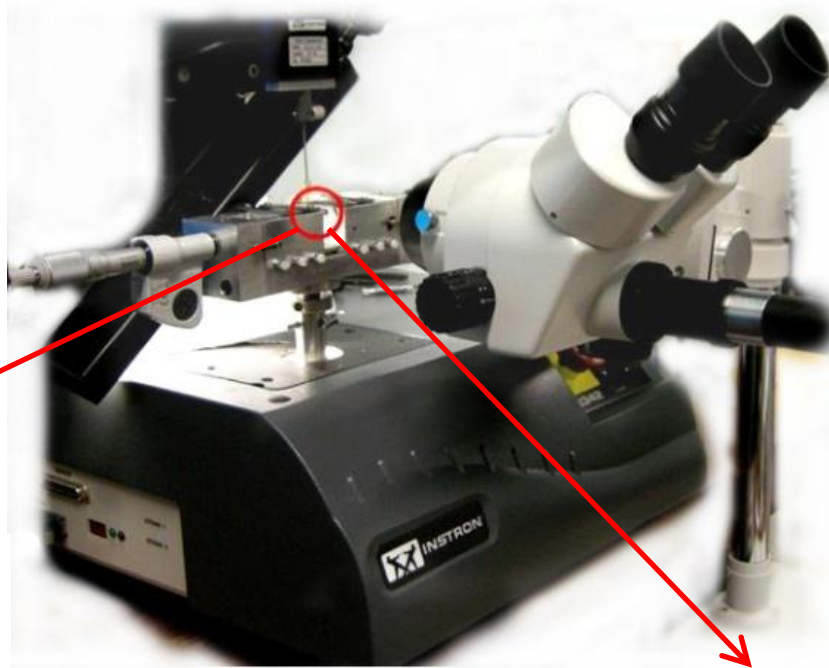
→ Pull and measure force to debond droplet (0.1-0.3 N)

→ Knife blades retain droplet

→ Droplet = 0.1-0.3 mm length

A simple concept – *with many pitfalls in the execution*

# Microbond Test for GF-PP IFSS



# Microbond Samples

Glass fibre



PP fibre



Inert Gas (N<sub>2</sub>)  
RT-220°C  
Isothermal 8 min  
Cool to ambient  
temperature



Solid  
polymer  
droplet

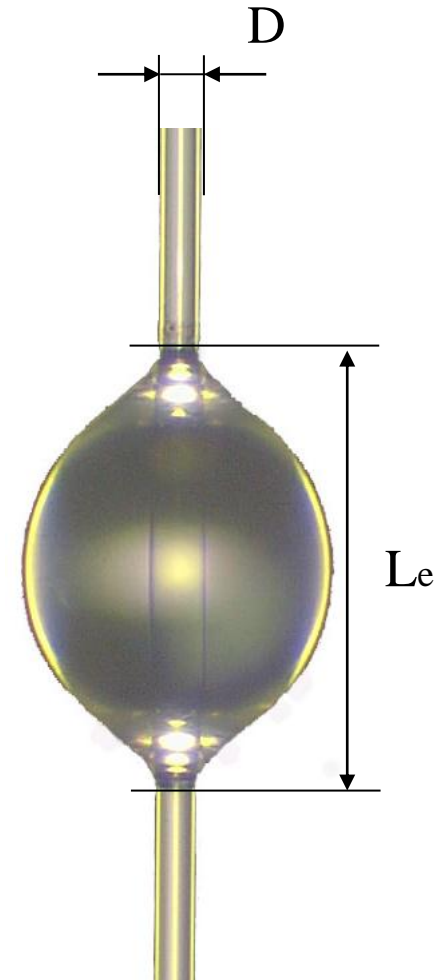
Glass fibre



Epoxy droplet

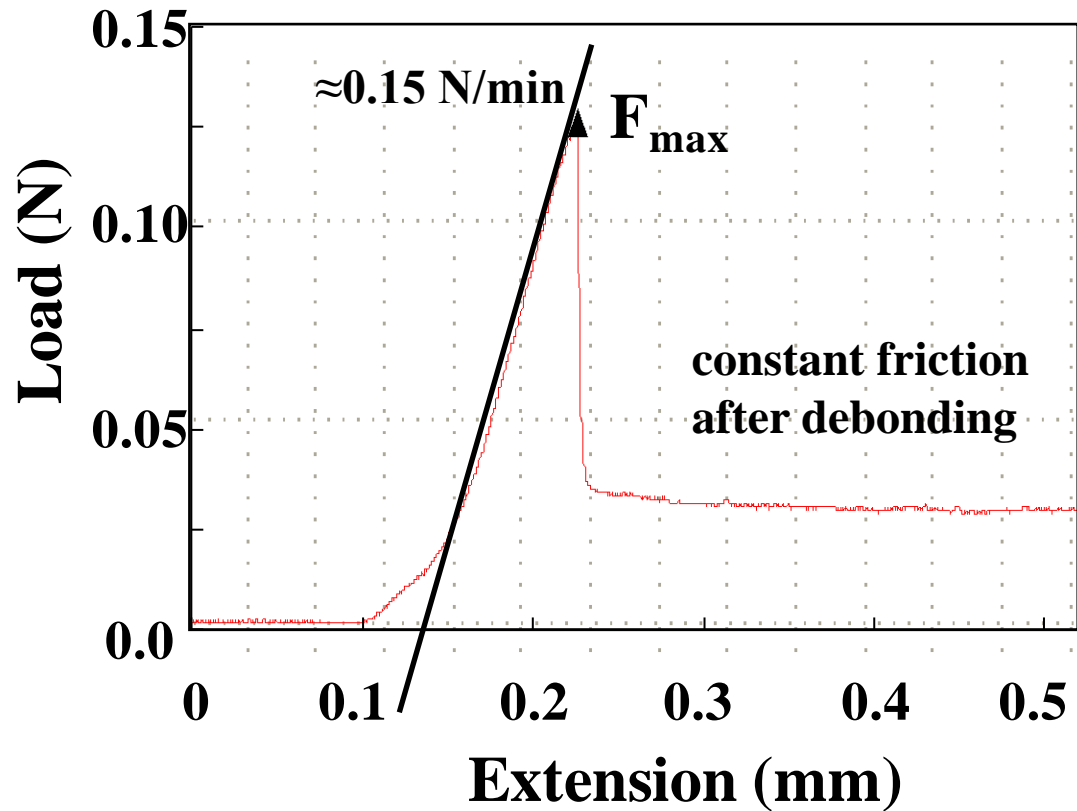
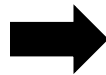
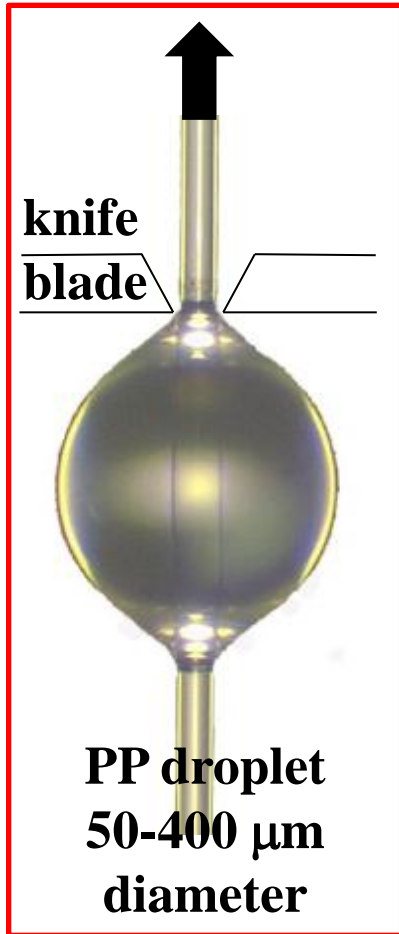


In Air  
1 hr at 60°C +  
2 hr at 120°C  
Cool to ambient inside  
the oven

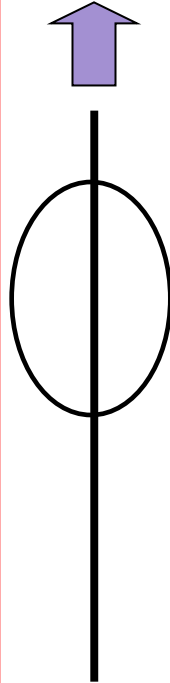


Embedded Interfacial  
Area:  $A_e = \pi \times D \times L_e$

# Microbond Test for GF-PP IFSS



Extension rate 0.1 mm/min

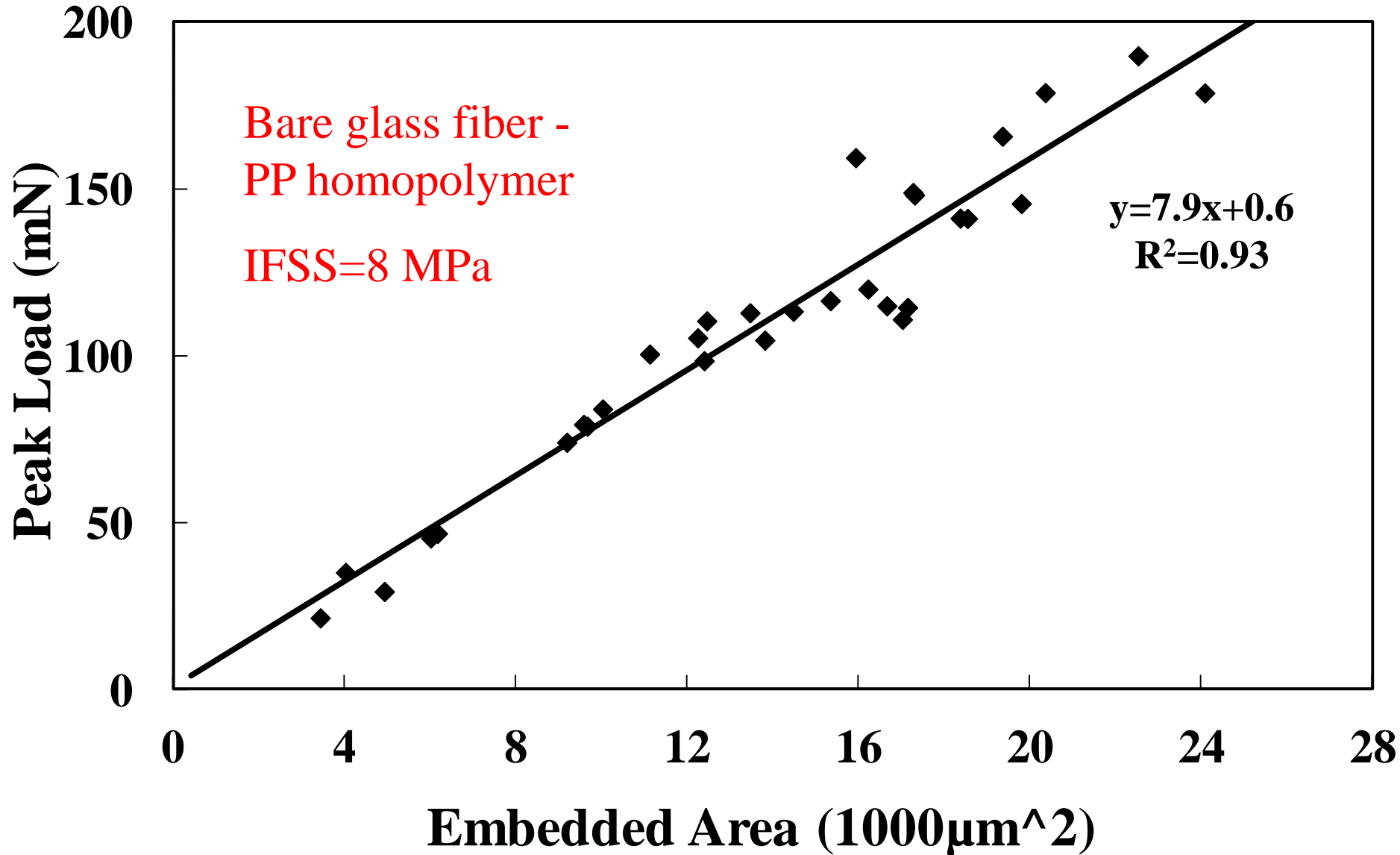


Assuming shear stress is distributed uniformly around the interface then -

**Average IFSS:**

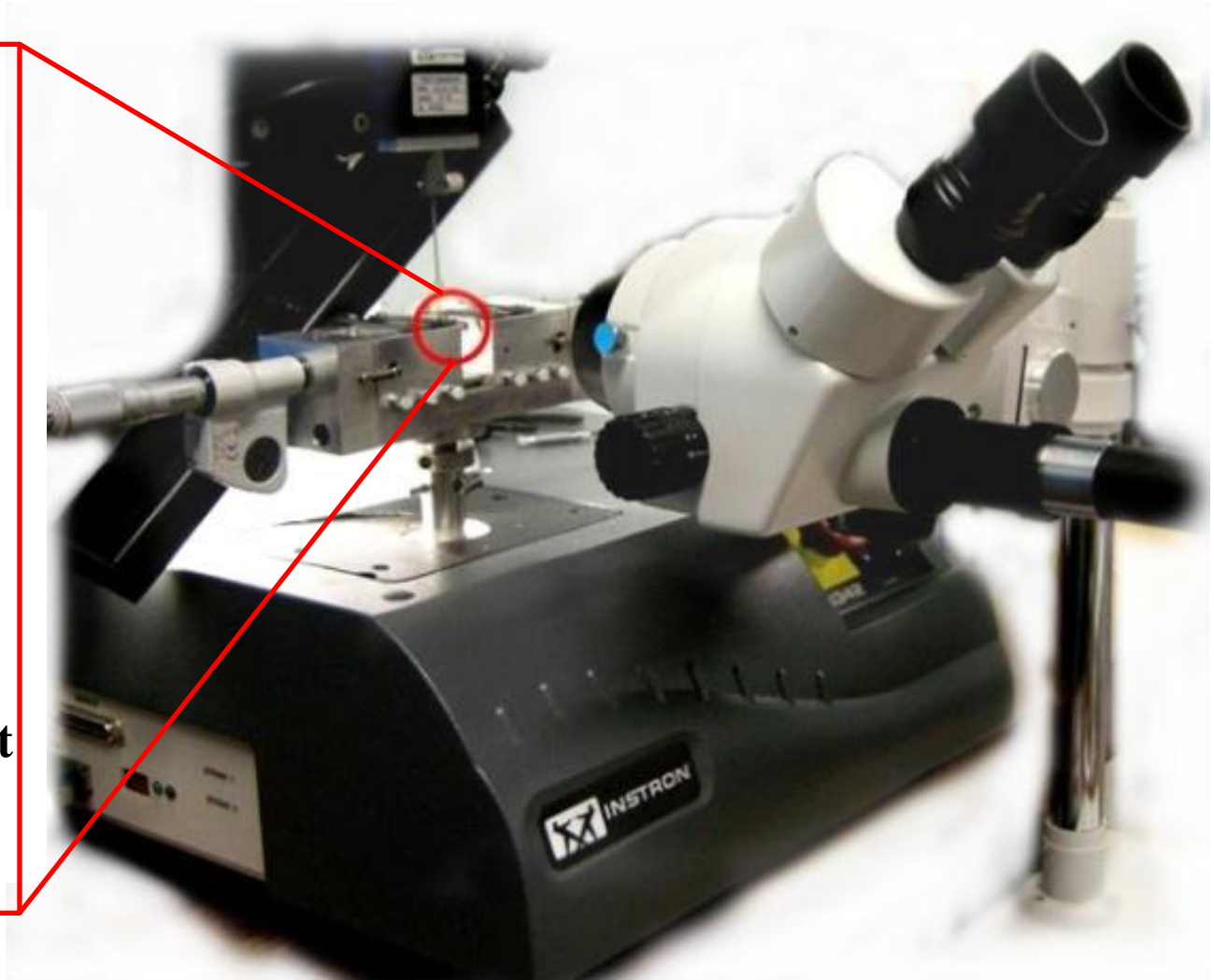
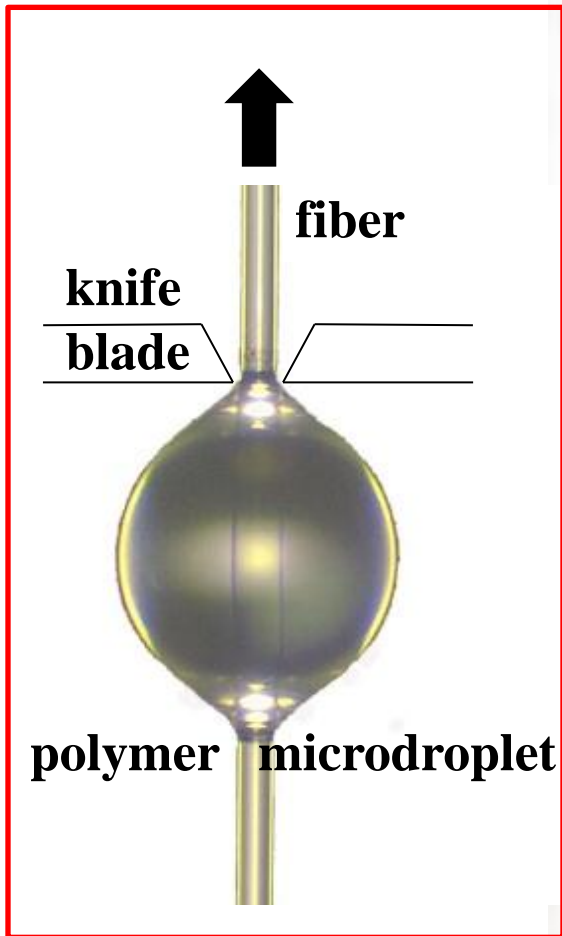
$$\tau = \frac{F_{\text{max}}}{A_e}$$

# PP-Glass IFSS by Microbond Method



Now need to run test at different temperatures

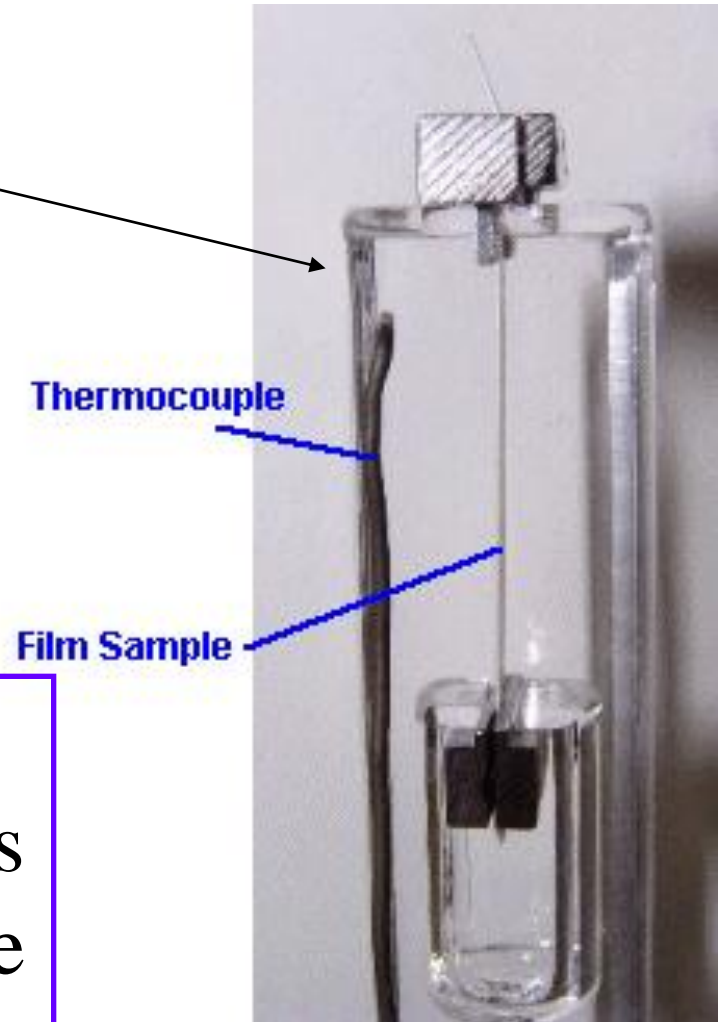
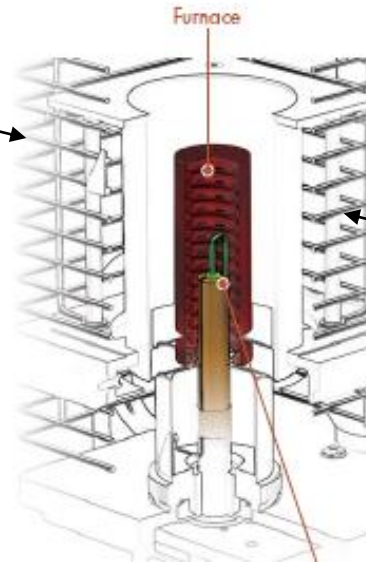
# Microbond Test for IFSS



Accurate test environment temperature control is a challenge

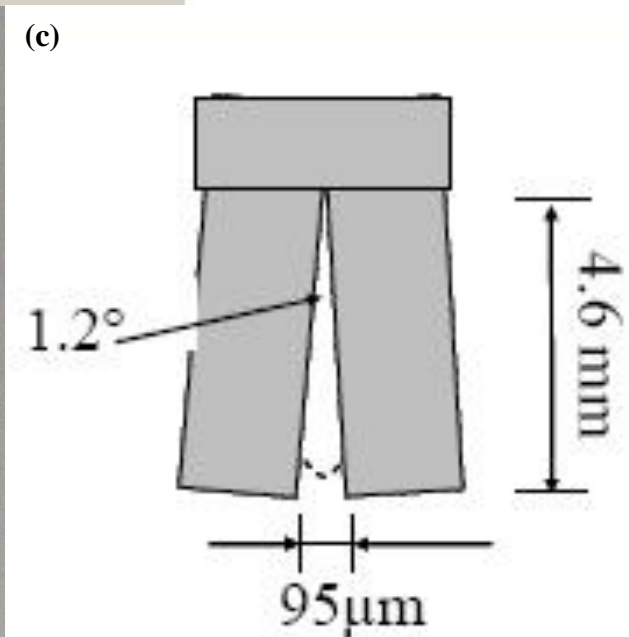
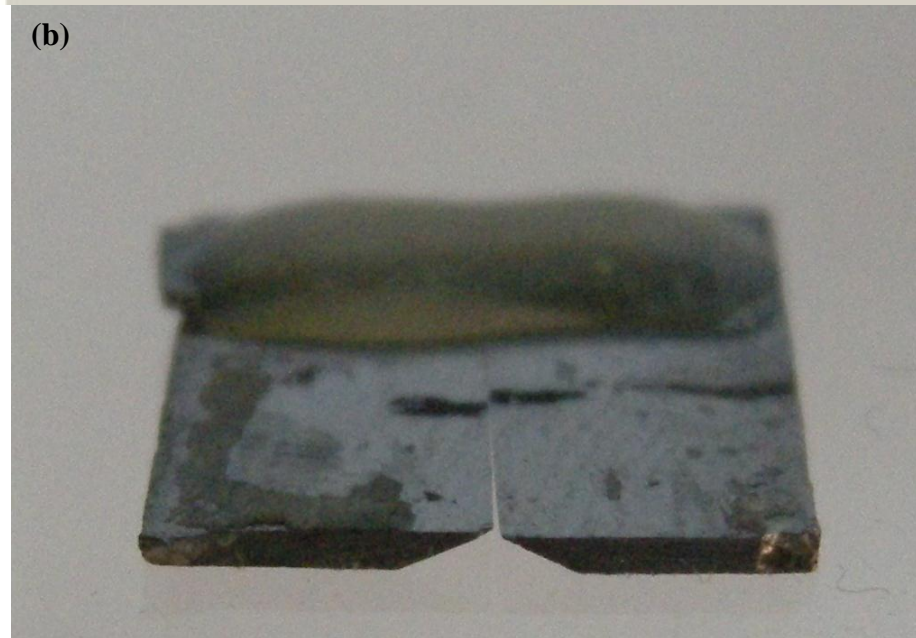
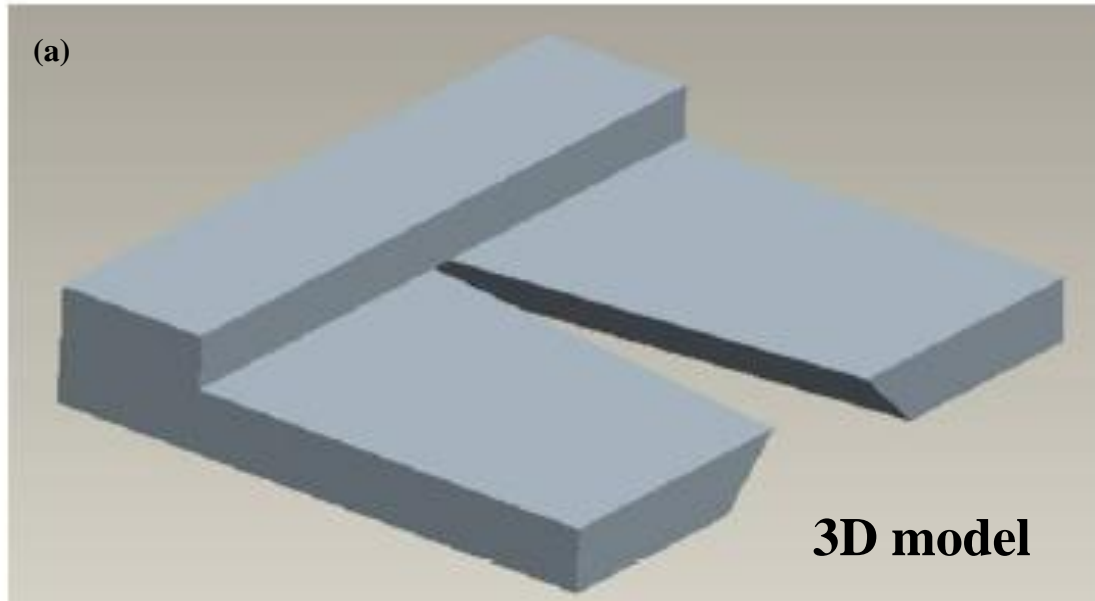


# Q400 EM TMA in Fiber/Film Mode



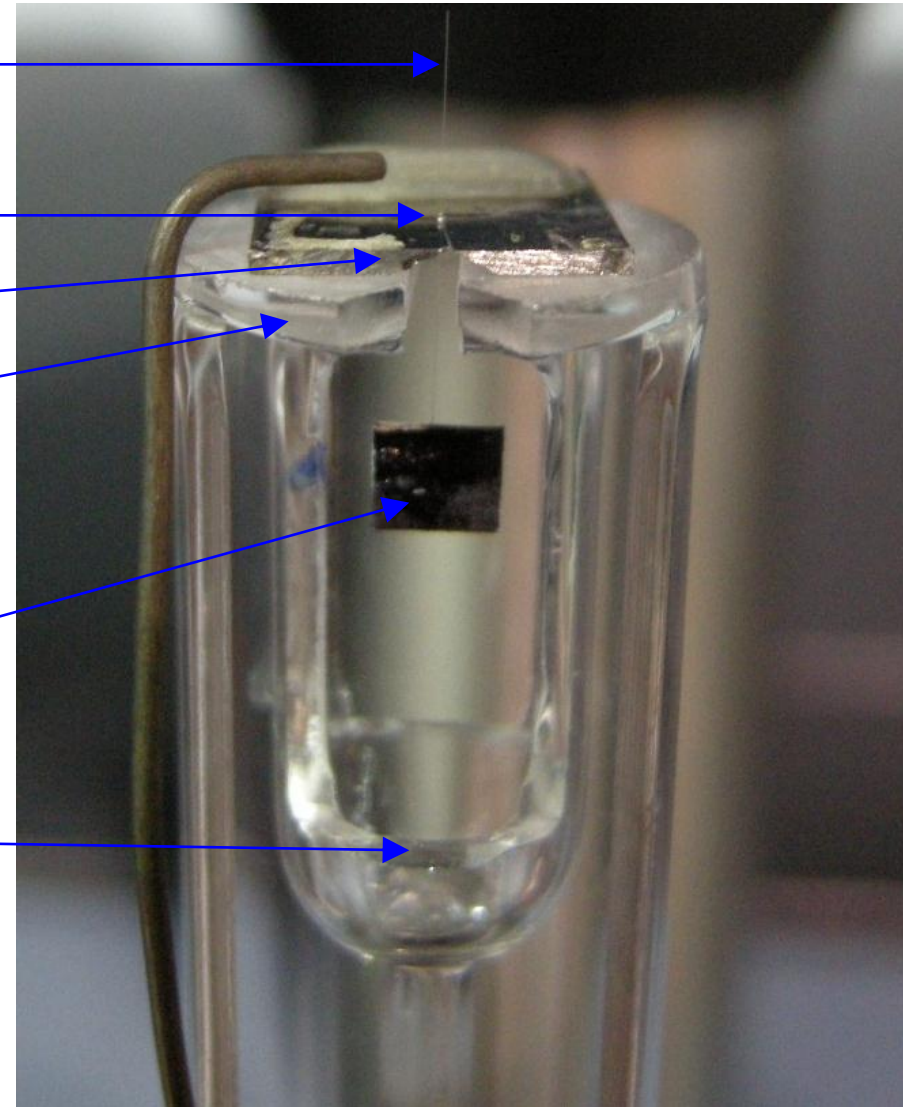
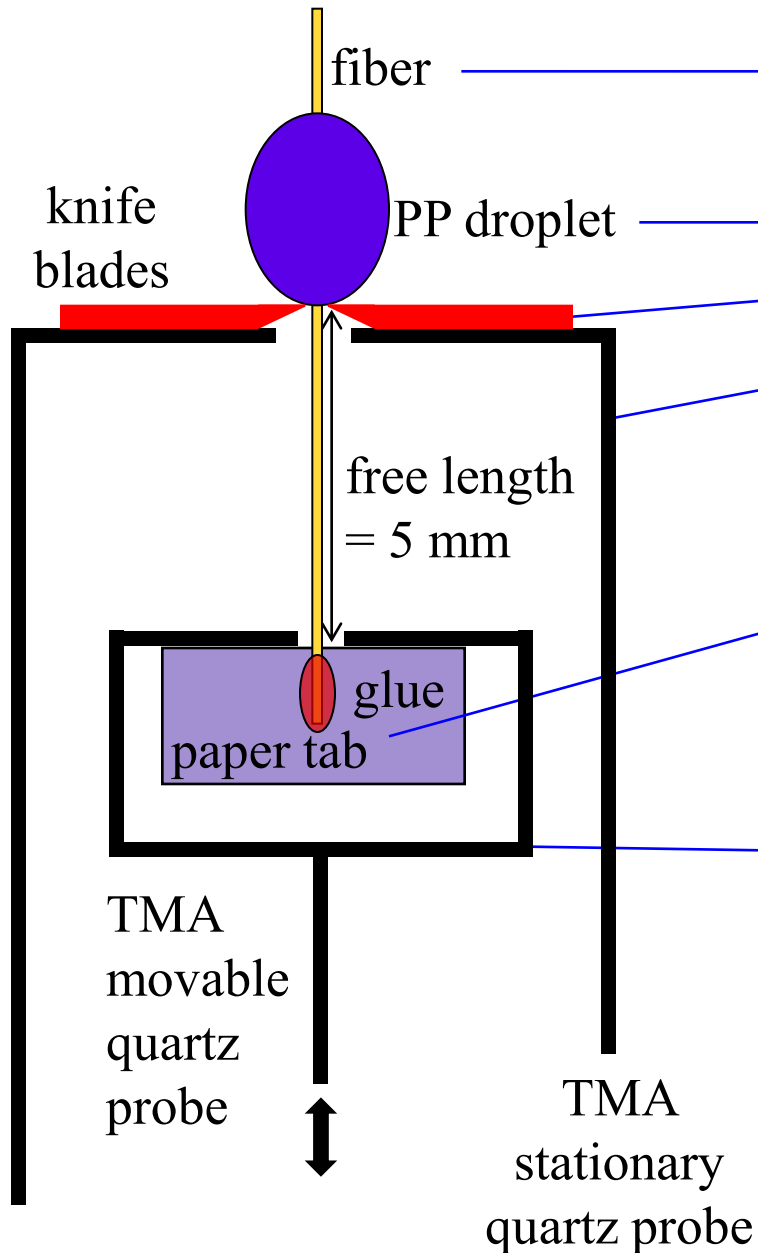
Strain-stress experiments  
possible on fiber/film samples  
in well controlled temperature  
environment

# Knife Blades for TMA Microbond





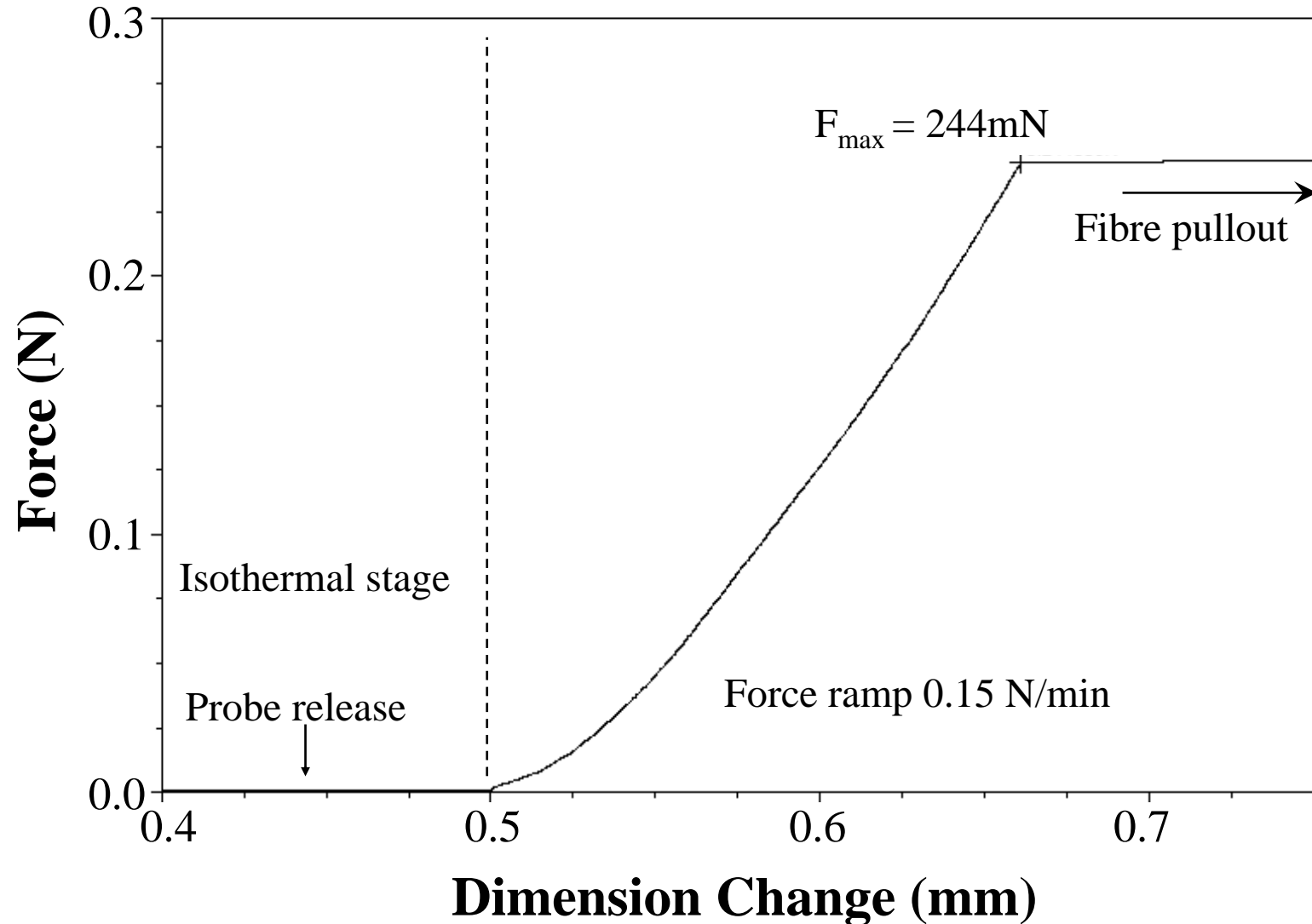
# TMA Microbond Test Configuration



# TMA Microbond Test

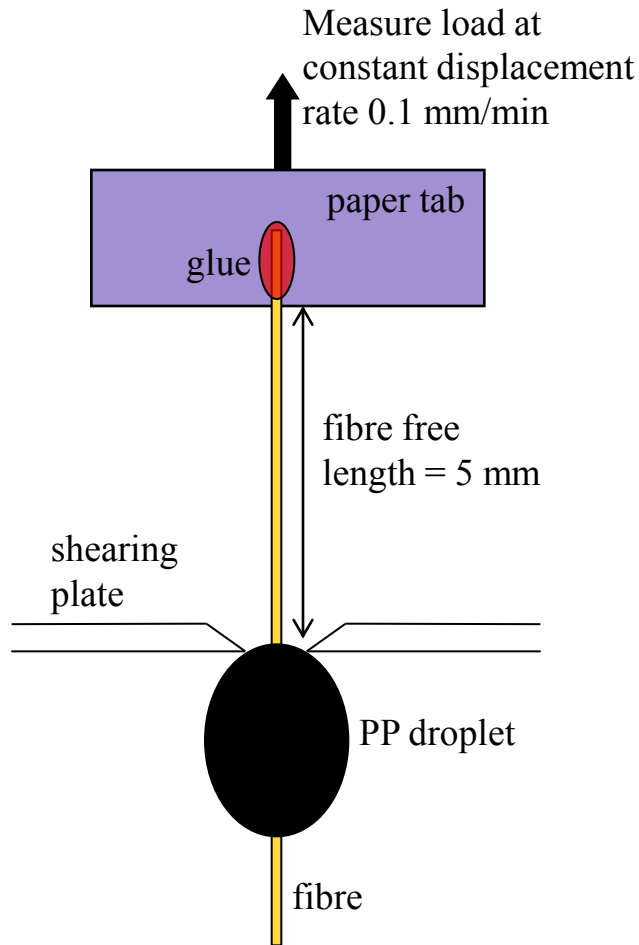
Isothermal at test temperature,

Ramp force (0.15 N/min) and measure displacement

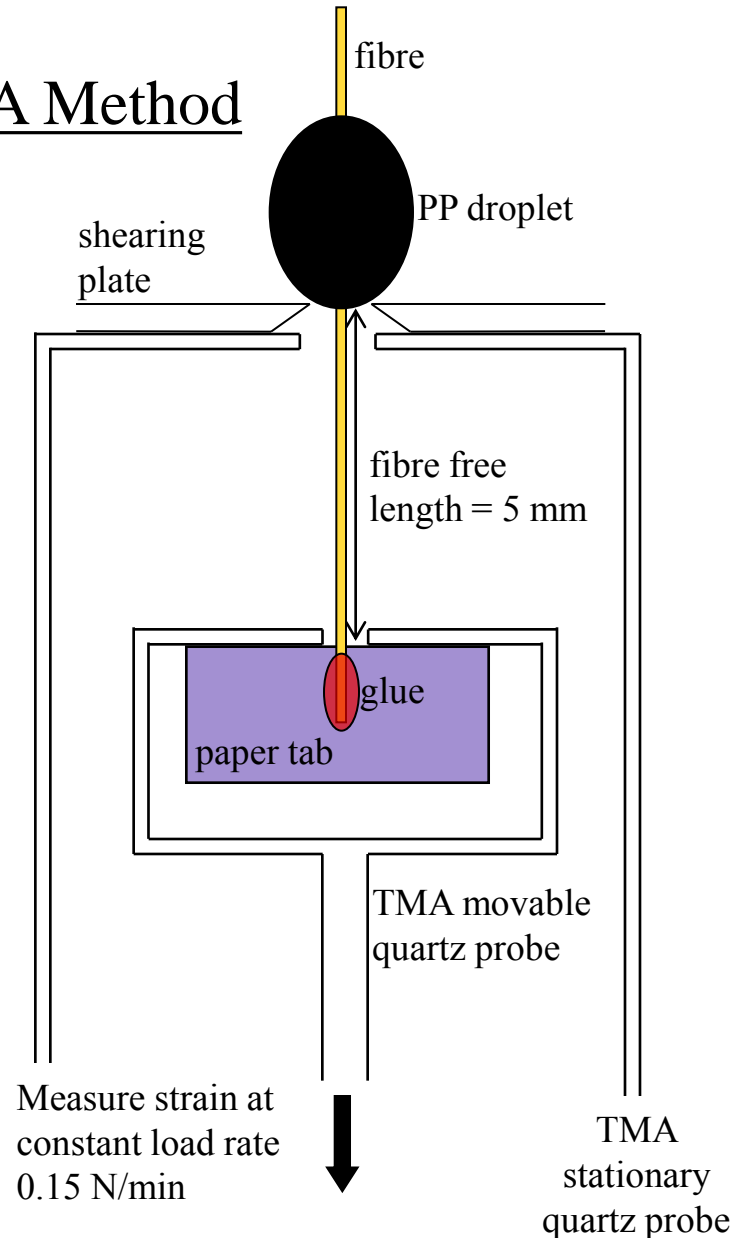


# Comparison Microbond Test Configurations

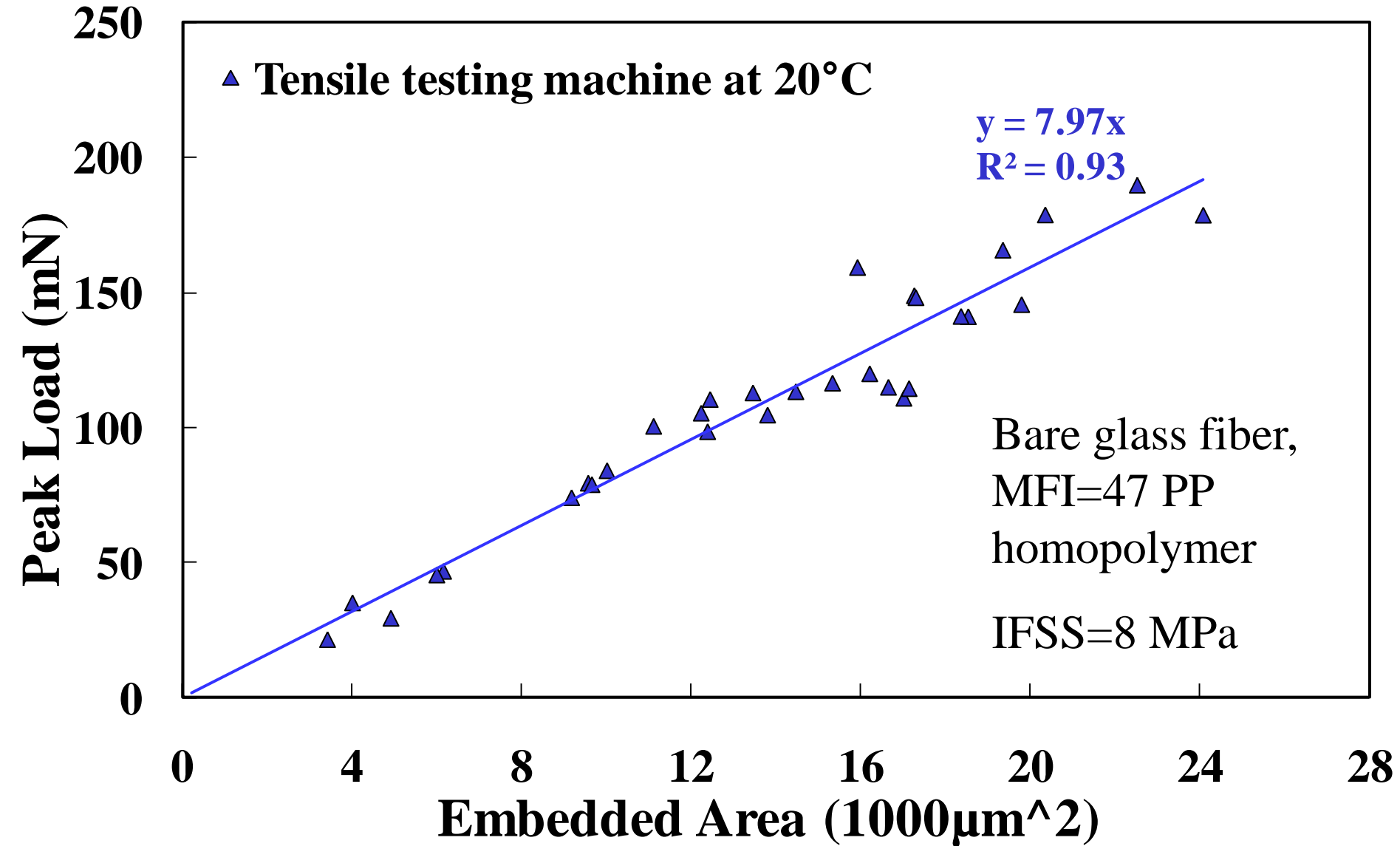
## “Standard” Method



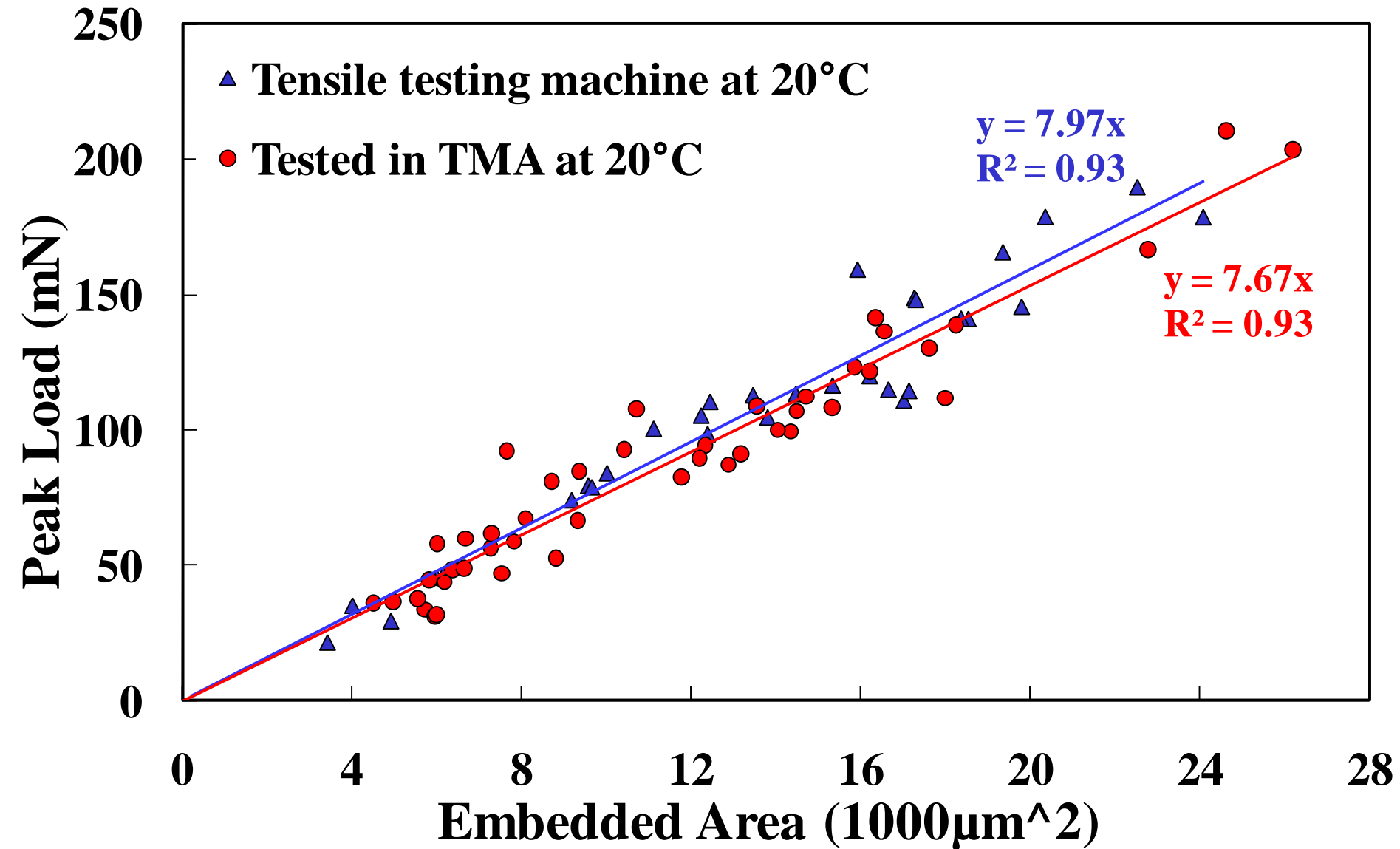
## TMA Method



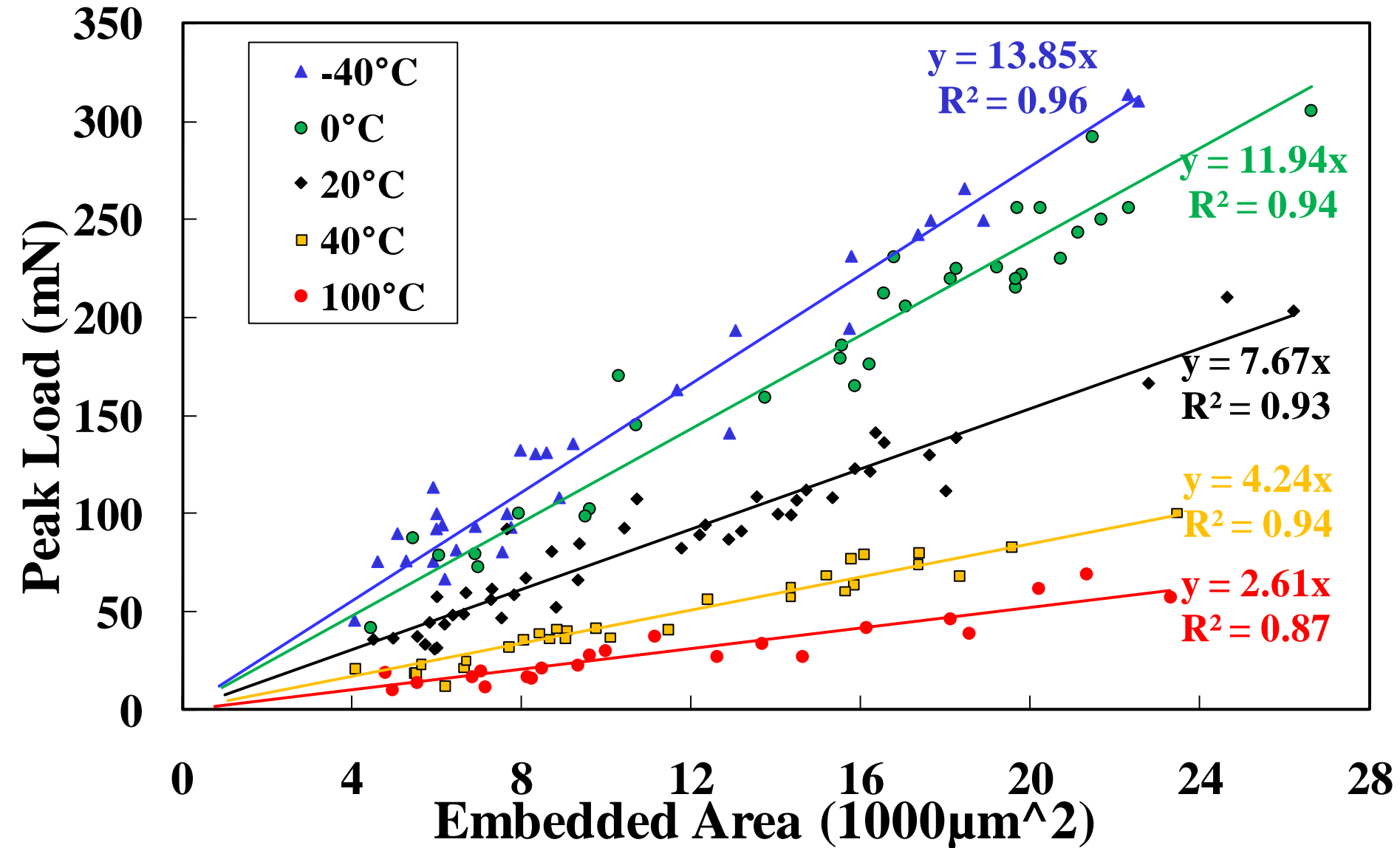
# PP-Glass IFSS by Microdroplet Method



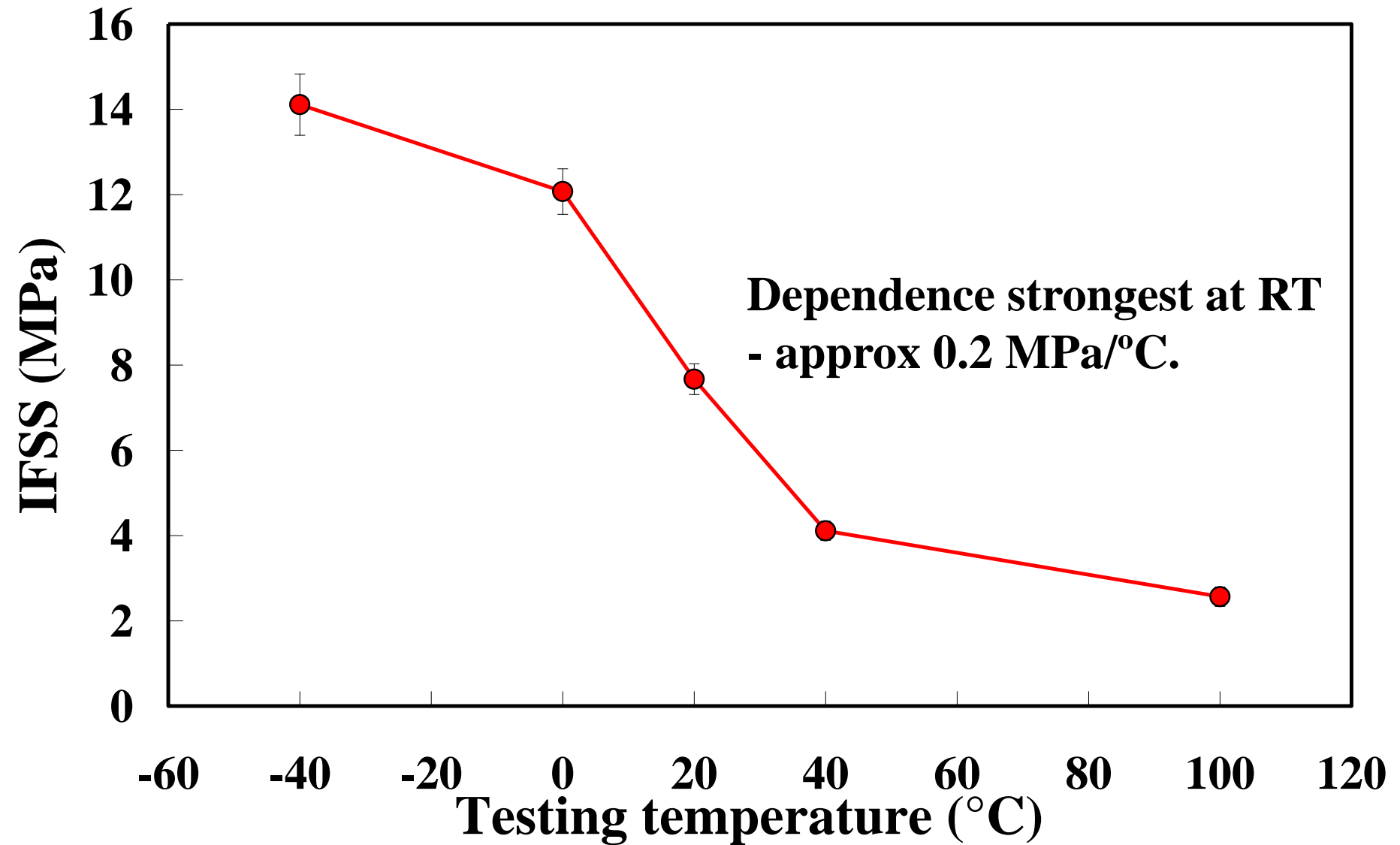
# PP-Glass IFSS by TMA Microdroplet Method



# PP-Glass IFSS by TMA Microbond Method



# PP-Glass IFSS vs Test Temperature



# Model Residual Thermal Stress at Interface

$$\sigma_{rm} = E_m (\alpha_m - \alpha_{fT}) \Delta T$$

$$\sigma_{rm} = A_1 (1 - b^2 / r^2) \text{ where } b = F(V_f)$$

$$\text{for } A_1 \text{ solve } \begin{bmatrix} X_{11} & X_{12} \\ X_{21} & X_{22} \end{bmatrix} \begin{bmatrix} A_1 \\ A_3 \end{bmatrix} = \begin{bmatrix} (\alpha_m - \alpha_{fL}) \Delta T \\ (\alpha_m - \alpha_{fT}) \Delta T \end{bmatrix}$$

$$\text{where } X_{11} = 2 \left( \frac{\nu_m}{E_m} + \frac{\nu_A}{E_L} \frac{V_m}{V_f} \right) \quad X_{12} = - \left( \frac{V_m}{E_L V_f} + \frac{1}{E_m} \right)$$

$$X_{21} = - \left( \frac{(1 - \nu_f) V_m}{E_m V_f} + \frac{(1 - \nu_m)}{E_m} + \frac{(1 + \nu_m)}{E_m V_f} \right) \quad X_{22} = \frac{X_{11}}{2}$$

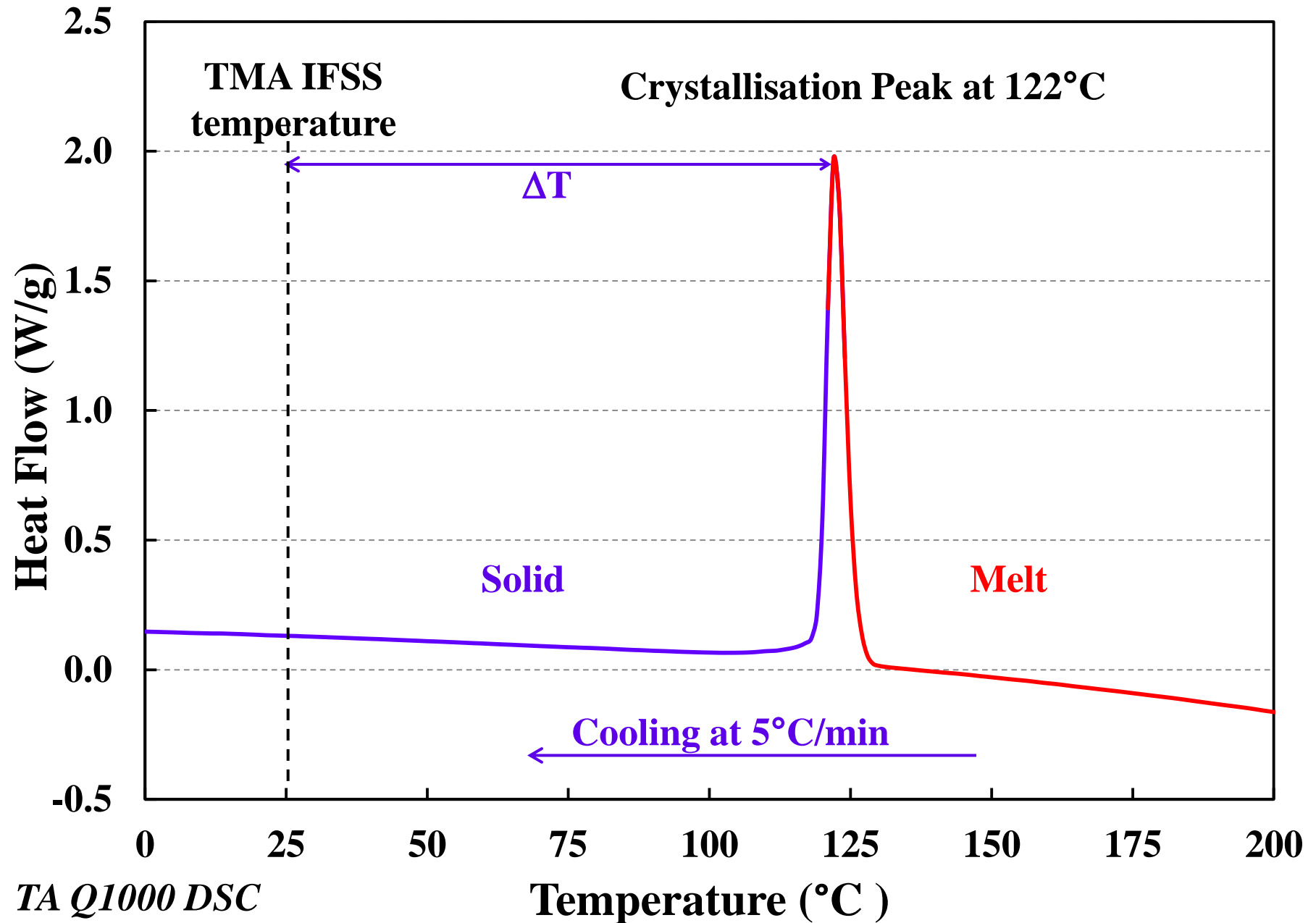
Nairn, J.A., Polymer Composites, 6, (1985) 123.

Wagner H.D. and Nairn J.A., Compos.Sci.Tech., 57, (1997) 1289

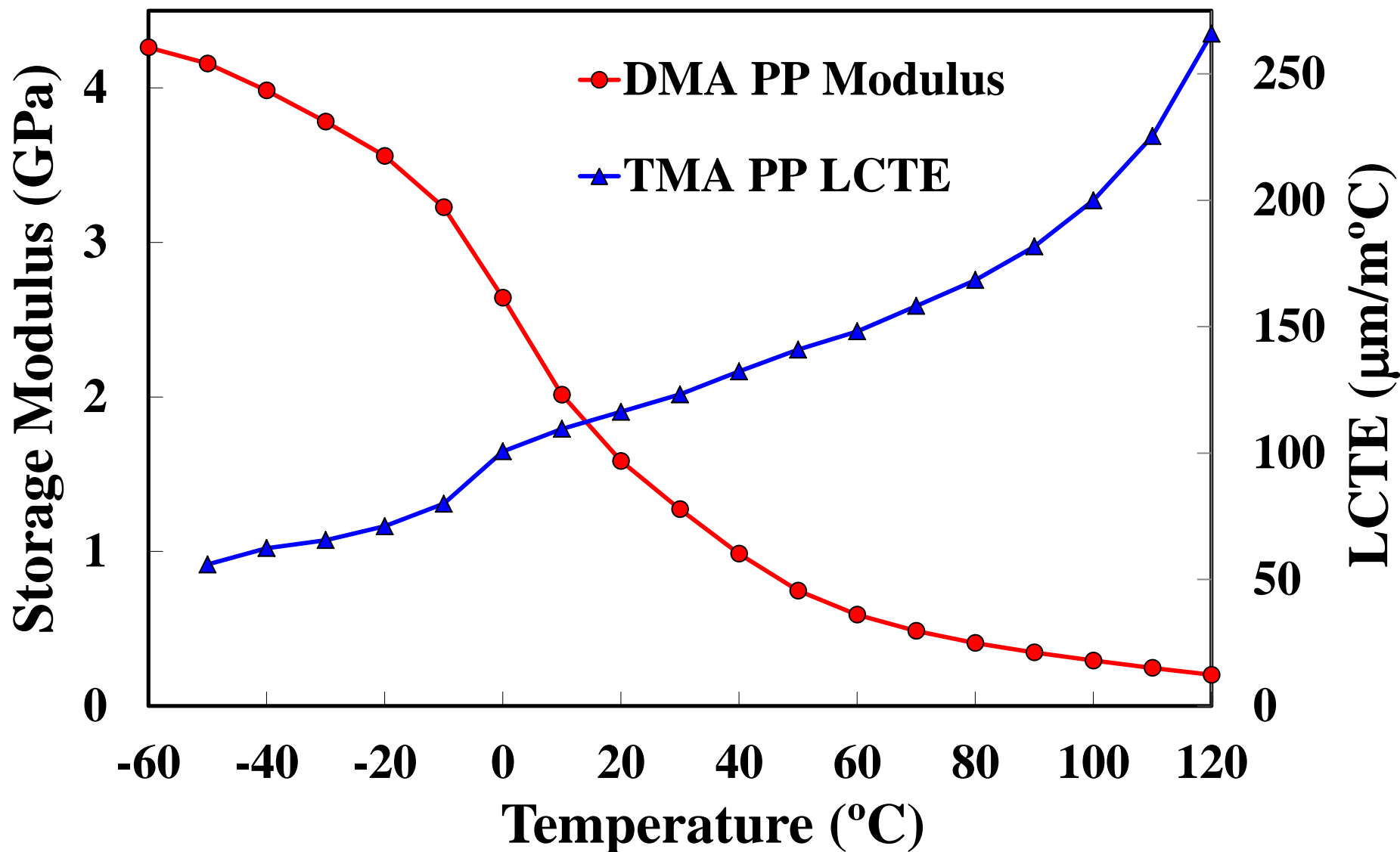
In both cases  $\sigma_{rm} \propto \Delta T \Rightarrow \underline{\text{IFSS}} \propto \Delta T$



# DSC Cooling Run for PP Solidification



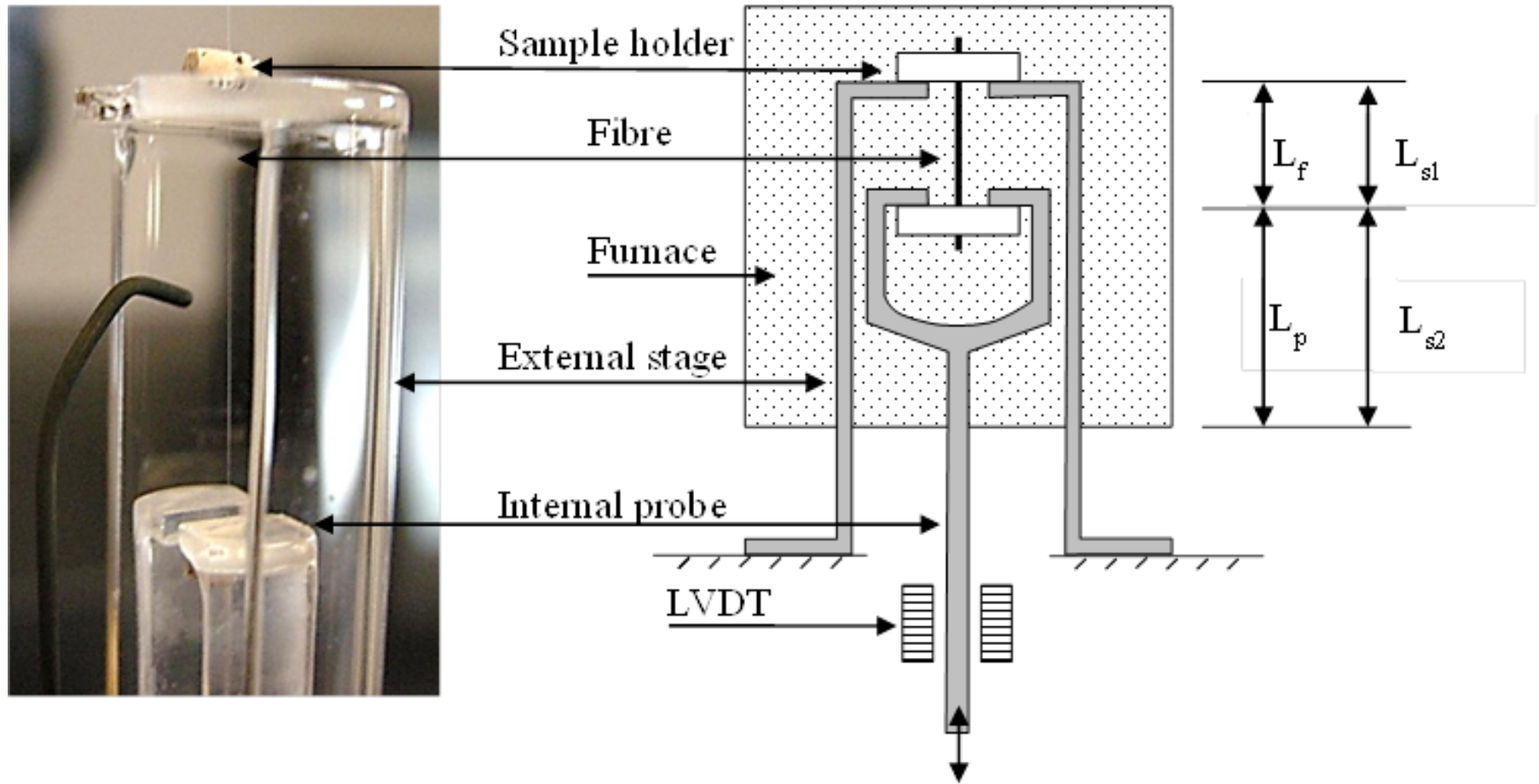
# Input for Residual Thermal Stress Model



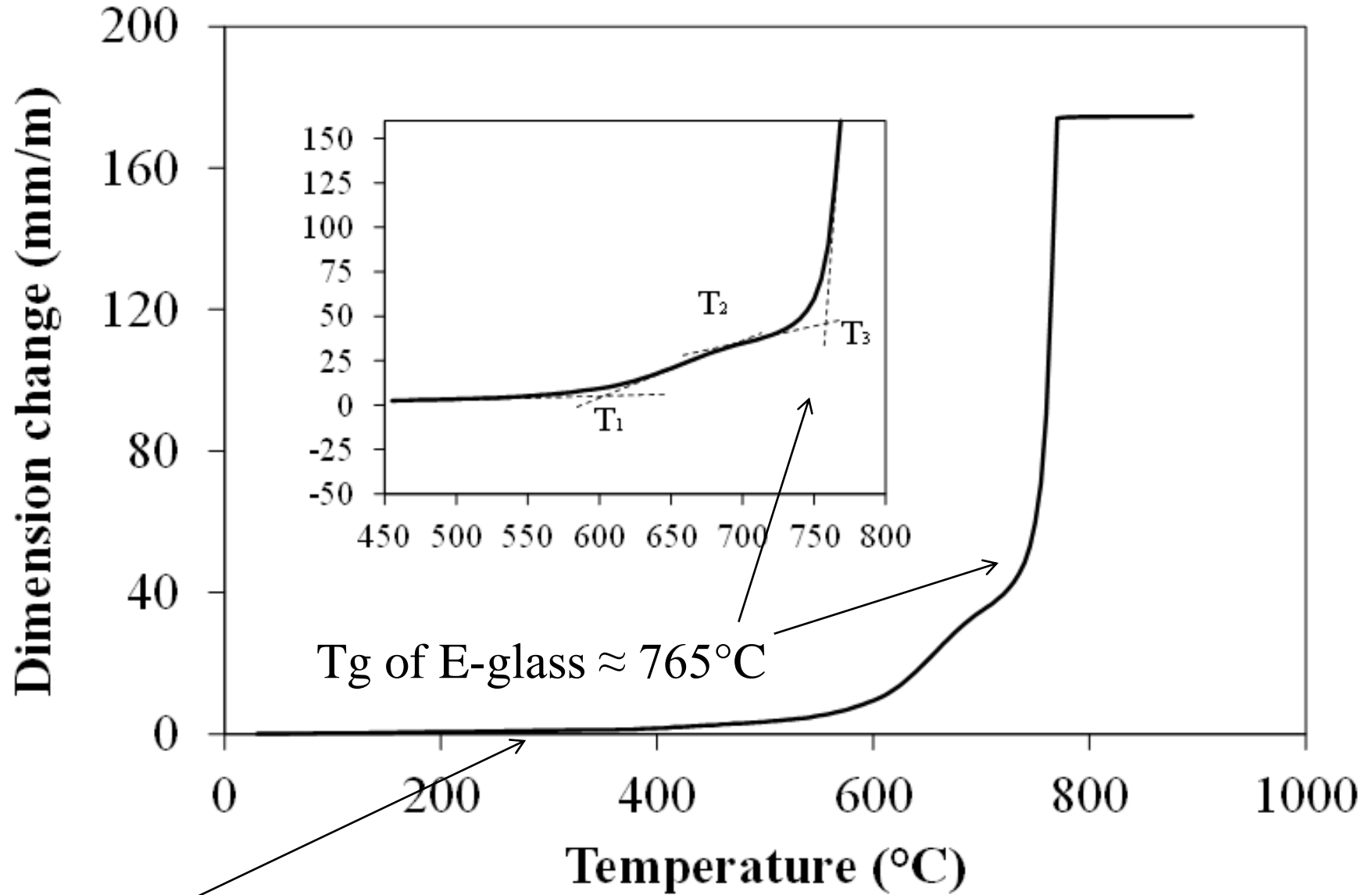
*TA Q800 DMA*

*TA Q400 TMA*

# TMA of Single Glass Fibre in Q400 EM

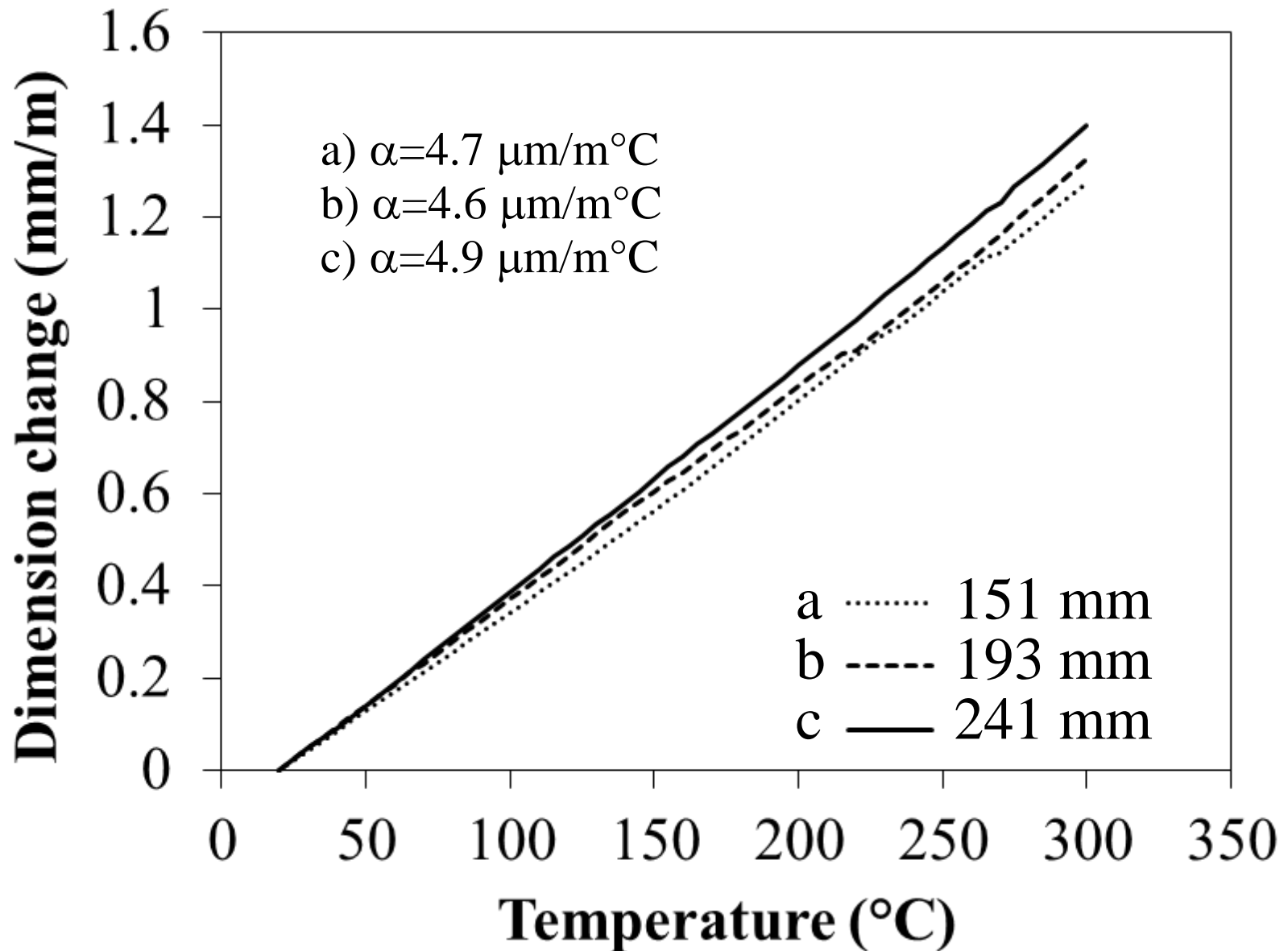


# TMA of Single Glass Fibre in Q400 EM

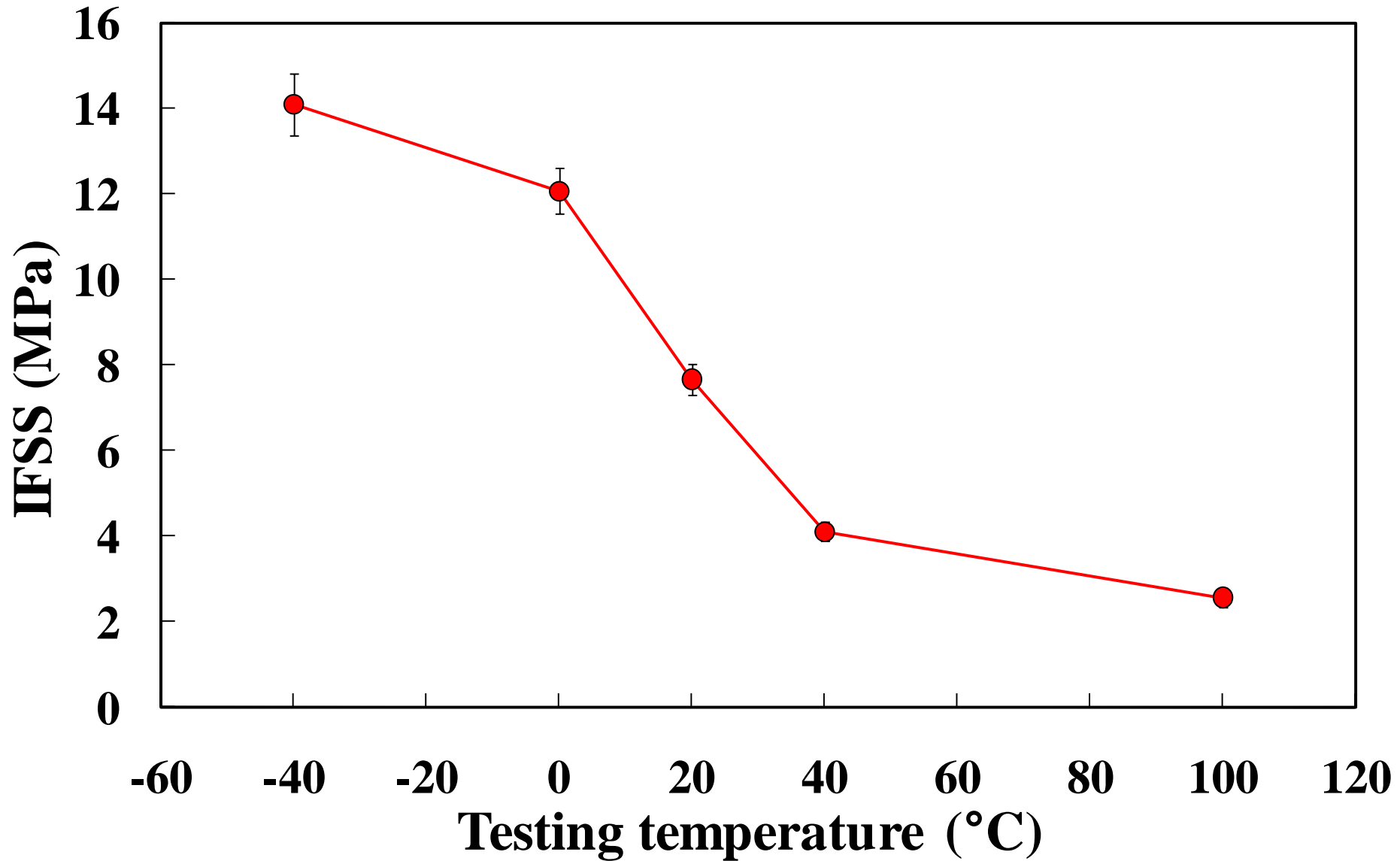


Linear region (23-300°C) for LCTE

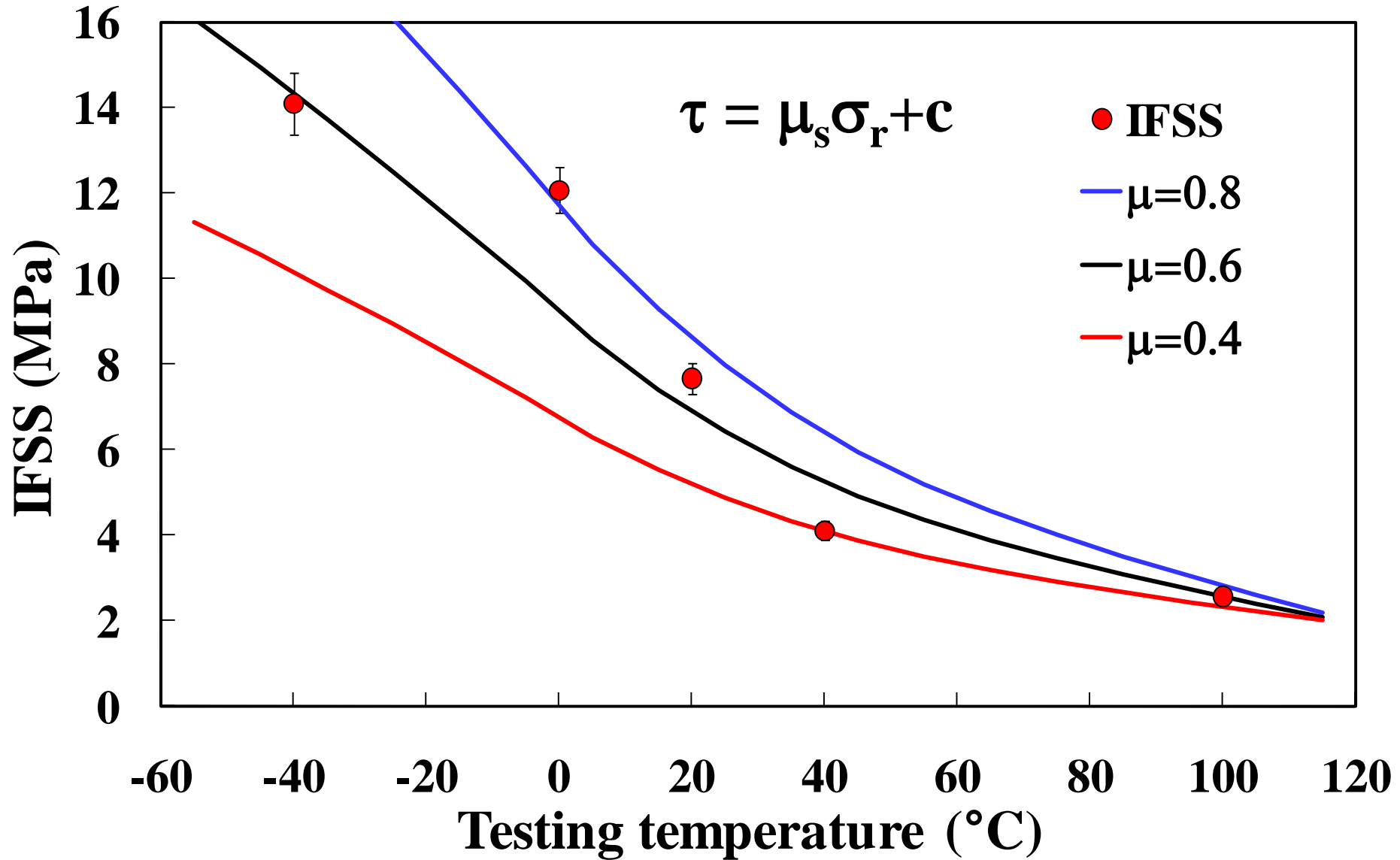
# LCTE of Single Glass Fibre in Q400 EM TMA



# PP-Glass IFSS vs Test Temperature



# PP-Glass IFSS vs Test Temperature



Schoolenberg 1995, GF-PP static coefficient of friction = 0.65

# Conclusions (GF-PP)

- Residual compressive stress at the composite interface may contribute significantly to the apparent IFSS
- Magnitude of these stresses is strongly influenced by **test temperature** vs the **solidification temperature**
- This results in an expectation that thermoplastic composite IFSS *should be* temperature dependent
- The TMA microbond test has enabled measurement of the IFSS of GF-PP from **-40°C** up to **100 °C**
- A strong dependence of GF-PP Interfacial Strength on Test Temperature has been observed

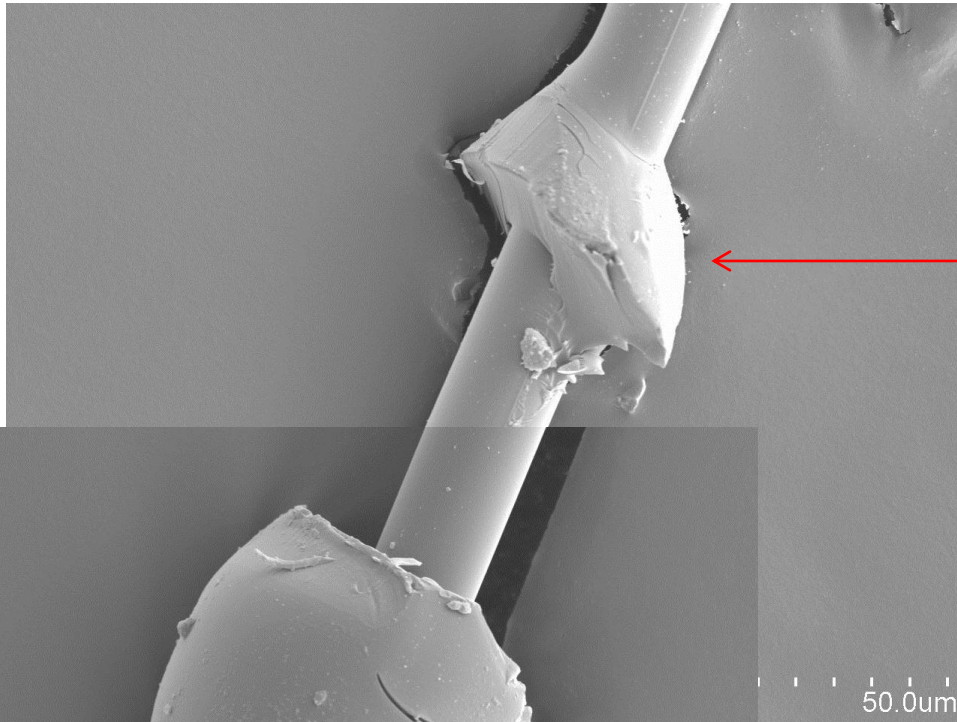


- **Residual compressive stress at the composite interface may contribute significantly to the apparent IFSS**
- Magnitude of these stresses is strongly influenced by sample temperature vs. curing temperature
- This results in an expectation that thermoplastic composite IFSS should be temperature dependent
- The TMA microbond test enabled measurement of the IFSS of GF-PP from -100 °C up to 100 °C
- **A strong dependence of GF-PP Interfacial Strength on Test Temperature has been observed**

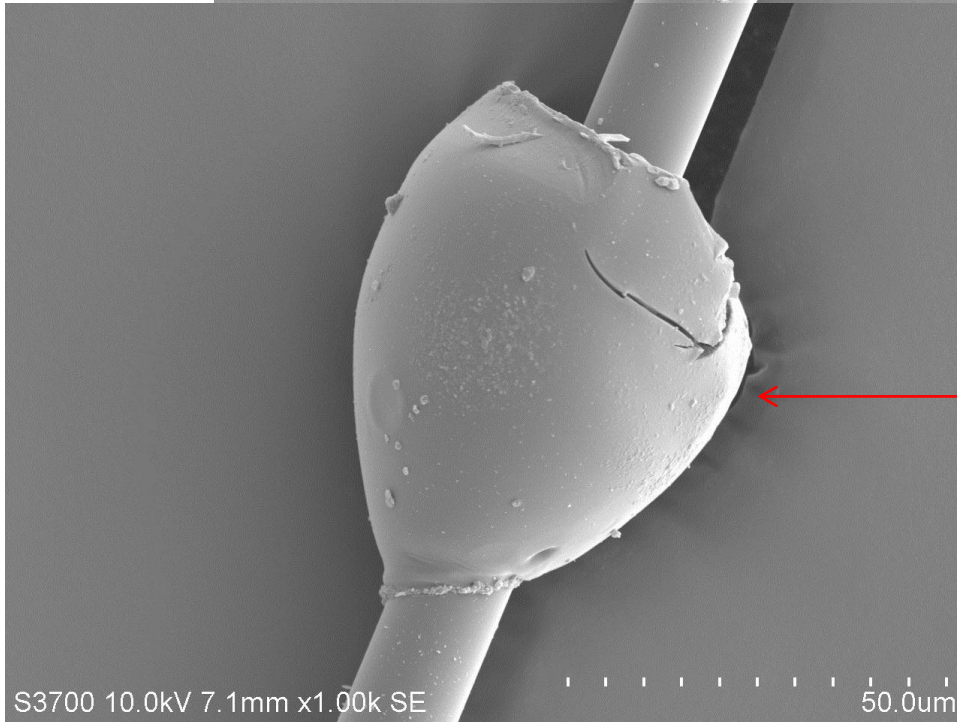
# **IFSS in Other Composite Systems ?**

- **IFSS correlates strongly with radial residual compressive stress in GF-PP**
- **GF-PP is a relatively low adhesion system**
- **What about other composite systems with high adhesion?**
  - **GF-Epoxy ?**
  - **GF-Polyamide ?**

# GF-Epoxy IFSS by TMA Microbond



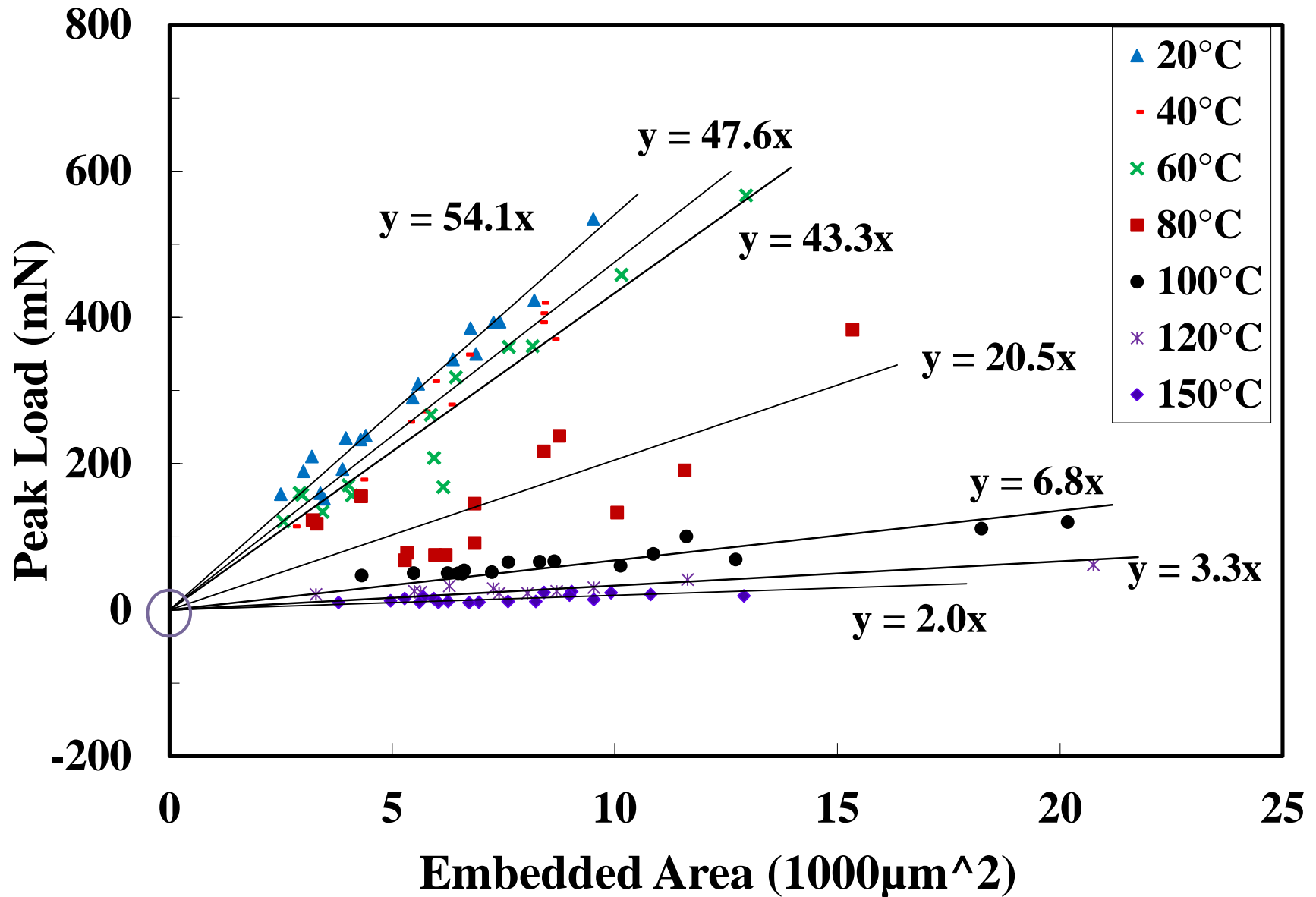
**Nondebonded part**



**Debonded part**

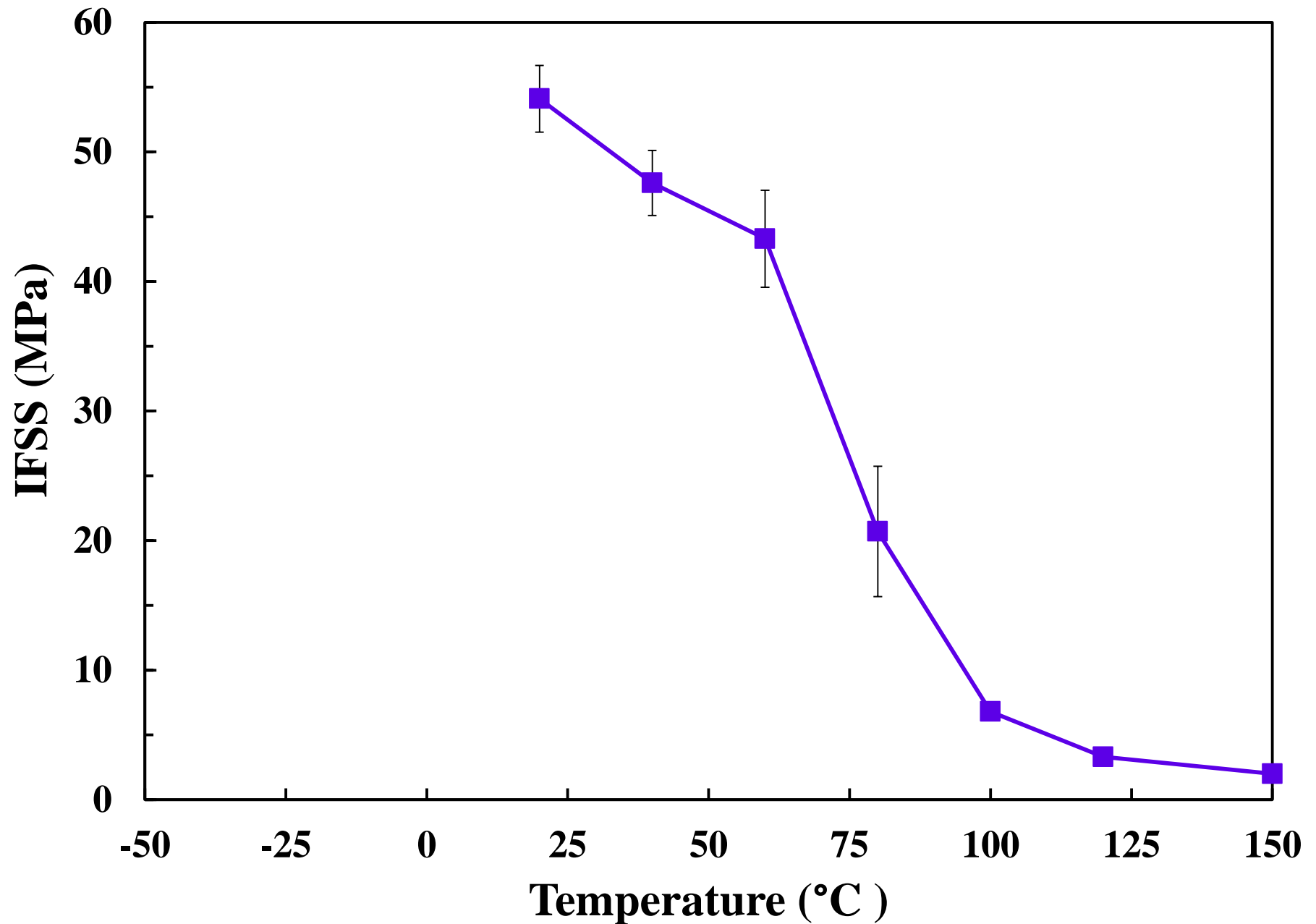
**Residual Le << Original Le**

# GF-Epoxy IFSS by TMA Microbond

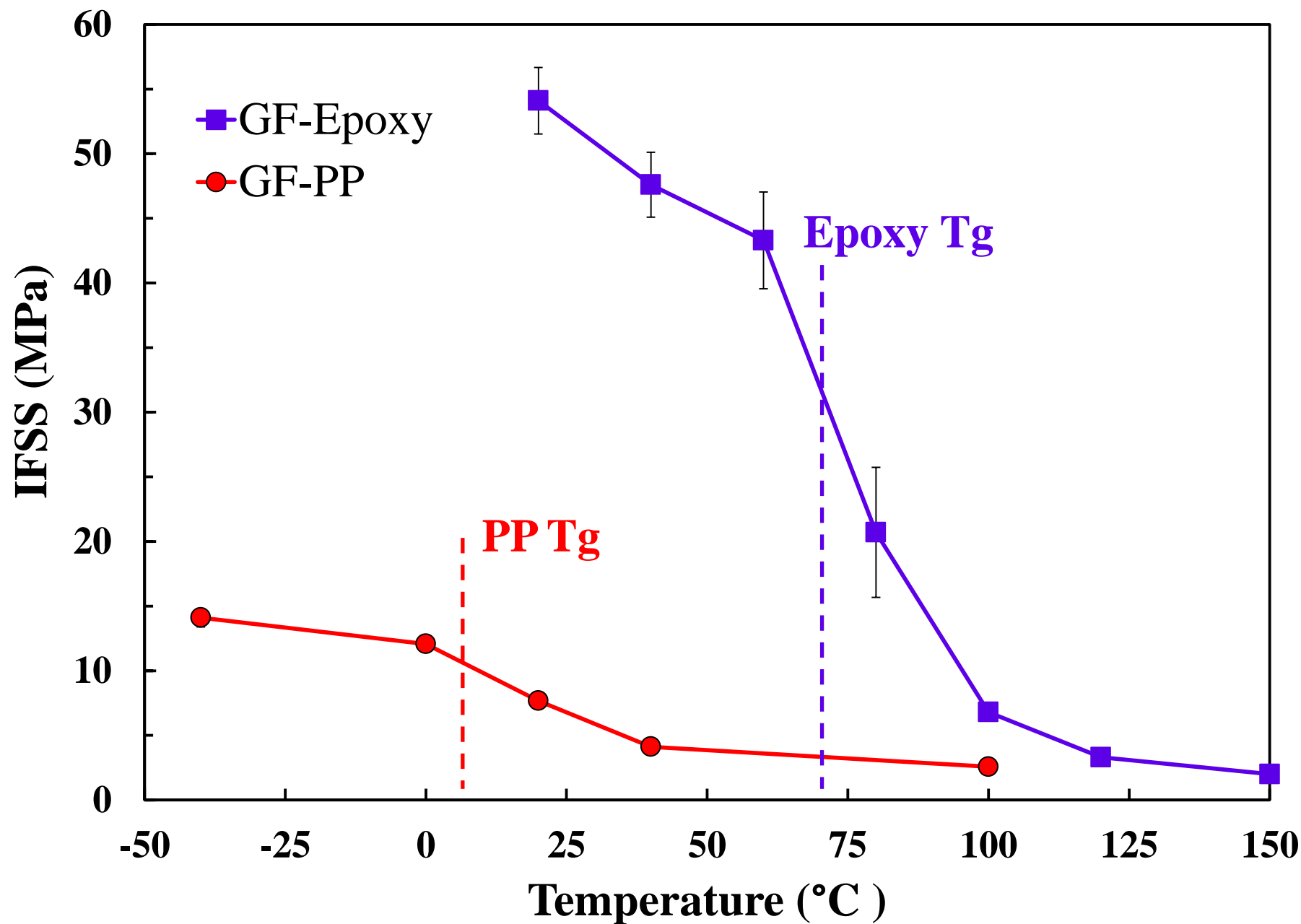


*Epoxy system DGEBA&TETA*

# GF-Epoxy IFSS



# GF-Polymer IFSS



# **Conclusions**

- **Thermal analysis equipment can provide an excellent temperature controlled environment in which to carry out micromechanical testing**
- **A strong dependence of Interfacial Shear Strength on Test Temperature has been observed in both GF-PP and GF-Epoxy Composites**
- **Residual compressive stress at the composite interface may contribute significantly to the apparent IFSS in GF-PP**

# Future Work

- **Adhesion in other Composite Systems**
  - Polyamide – Glass Fibre
  - Epoxy – Carbon Fibre
- **Test Development**
  - Microbond in the Q800 DMA ?
  - Microbond-DMA vs Temperature & Humidity